DESIGN, DEVELOPMENT AND FABRICATION OF THERMAL MEASURING SYSTEMS

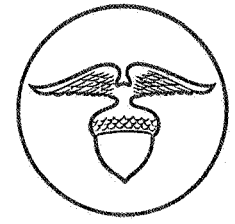
CONTRACT NO. NAS 8-11708

25 JUNE 1964-25 MARCH 1965

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
GEORGE C. MARSHALL SPACE FLIGHT CENTER
HUNTSVILLE, ALABAMA

I.A.BLACK

A.E.WECHSLER



Arthur D.Little,Inc.

DESIGN, DEVELOPMENT, AND FABRICATION OF THERMAL MEASURING SYSTEMS

FINAL REPORT

Contract No. NAS8-11708
25 June 1964 - 25 March 1965

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
GEORGE C. MARSHALL SPACE FLIGHT CENTER
HUNTSVILLE, ALABAMA

Prepared by

ARTHUR D. LITTLE, INC.
CAMBRIDGE, MASSACHUSETTS

I. A. Black
A. E. Wechsler

TABLE OF CONTENTS

		Page
1.0	GUARDED COLD PLATE THERMAL CONDUCTIVITY APPARATUS	1
1.1	INTRODUCTION	1
1.2	CAPABILITIES OF THE CALORIMETER	3
1.3	DESCRIPTION OF THE CALORIMETER	4
1.3.1	Measuring & Guard Vessels (Cold Plate Assembly)	4
1.3.2	Warm Plate Assembly	10
1.3.3	Sample Chamber	14
1.3.4	Bell Jar	17
1.3.5	Base Plate	19
1.4	INSTRUMENTATION	21
1.4.1	Vacuum Pumping System	21
1.4.2	Measurement of Vacuum in the Bell Jar and the Sample Chamber	21
1.4.3	Measurement and Control of the Warm Plate Temperature	24
1.4.4	Measurement of Cryogen's Boil-Off Rate	26
1.4.5	Measurement and Control of Cryogen's Pressure	28
1.4.6	Measurement and Control of Warm Plate's Vertical Position	29
1.4.7	Bell Jar and Cold Plate Lifting Hoist	31
1.5	INSTALLATION INSTRUCTIONS	33
1.5.1	Position of the Calorimeter	33
1.5.2	Service of the Constant Temperature Oil Bath	33
1.5.3	Water and Drain Requirements	33
1.5.4	Electrical Requirements	33

TABLE OF CONTENTS (cont'd)

		Page
1.5.5	Requirement for Compressed Gases	35
1.6	PROCEDURE FOR CHANGE OF TEST SAMPLE	35
1.6.1	Disassembly of the Calorimeter	35
1.6.2	Reassembly of the Calorimeter	38
1.7	TEST PROCEDURE	42
1.7.1	Evacuation of the Bell Jar and Sample Chamber	42
1.7.1.1	High Vacuum in the Bell Jar and in the Sample Chamber	42
1.7.1.2	High Vacuum in the Bell Jar and Pressure Other than High Vacuum in the Sample Chamber	45
1.7.2	Adjusting of the Warm Plate Temperature	45
1.7.3	Filling the Guard and Measuring Vessels with Cryogen	45
1.7.4	Taking Data	47
1.7.5	Removal of the Cryogen from the Calorimeter and Warm-Up Procedure	48
1.7.6	Shutdown of the Vacuum System	49
1.7.6.1	Complete Shutdown	49
1.7.7	Storing of the Calorimeter	50
1.8	APPENDIX	51
1.8.1	Data Sheet for Use with ADL Model-6 Calorimeter	51
1.8.2	Sample Calculations	54
1.8.3	Partial Listing of Purchased Components for the ADL Model-6 Calorimeter	59
1.8.4	List of Spare Parts	61

TABLE OF CONTENTS (cont'd)

		Page
2.0	THE LINE HEAT SOURCE APPARATUS AND THE THERMAL CONDUCTIVITY PROBE	63
	2.1 HISTORICAL REVIEW	63
	2.2 THEORETICAL CONSIDERATIONS	64
	2.2.1 The Line Heat Source	64
	2.2.2 The Thermal Conductivity Probe	66
	2.3 DESCRIPTION OF APPARATUS	68
	2.3.1 Line Heat Source Apparatus	68
	2.3.2 Thermal Conductivity Probe Apparatus	73
	2.3.3 Base Plate and Bell Jar Assembly	75
	2.4 INSTRUMENTATION	75
	2.5 OPERATING INSTRUCTIONS	78
	2.5.1 Line Heat Source Apparatus	78
	2.5.2 Thermal Conductivity Probe Apparatus	85
	2.6 SPARE AND REPLACEMENT PARTS	87
	2.7 SAMPLE CALCULATIONS	88
	2.7.1 Thermal Conductivity Probe	89
	2.7.2 Line Heat Source	91
3.0	REFERENCES	92
4.0	ACKNOWLEDGMENTS	94

LIST OF FIGURES

		Page
1	General View of the ADL Model-6 Calorimeter	5
2	ADL Model-6 Calorimeter (Cross Section)	6
3	Measuring and Guard Vessels (Cold Plate Assembly)	7
4	Warm Plate, Sample Chamber and Base Plate	11
5	Location of the Thermocouples in the Warm Plate	12
6	Warm Plate Heating Coil	13
7	Teflon Bearing	15
8	Sample Chamber	16
9	Bell Jar	18
10	Base Plate Assembly	20
11	Instrumentation	22
12	Vacuum System	23
13	Right Side Control Panel	25
14	Low Boil-Off Rate Meter	27
15	Adjustment Mechanism for the Warm Plate Position	30
16	Front Control Panel	32
17	City Water and Drainage Connections for the Hoist	34
18	Wiring Diagram	36
19	Guide to Assembly and Disassembly of the Calorimeter	37
20	Lifting of the Bell Jar	39
21	Lifting of the Cold Plate Assembly	40
22	Vacuum Control Valves for the High Vacuum Pumping Station	44
23	Height of the Oil in the Graduate at V_1 = 200 cm^3 and 2000 cm^3	55

LIST OF FIGURES (cont'd)

		Page
24	Line Heat Source Apparatus in the Sample Holder	69
25	Line Heat Source Apparatus	70
26	Constant Temperature Line Heat Source Holder	72
27	Schematic Diagram of Thermal Conductivity Probe	74
28	Thermal Conductivity Probe and Sample Holder	76
29	Schematic Diagram of Base Plate Assembly	77
30	Schematic Diagram of Instrumentation for Probe or Line Heat Source Apparatus	79
31	Thermocouple Wiring Diagram for Probe or Line Heat Source	80
32	Typical Data from Thermal Conductivity Probe	90

1.0 GUARDED COLD PLATE THERMAL CONDUCTIVITY APPARATUS

1.1 INTRODUCTION

The first 6-inch diameter calorimeter was designed and built approximately seven years ago. It is described in detail in the paper "Single Plate Apparatus for Tests of Low-Temperature Thermal Conductivity".[1] Since that time, it has been in constant use in our laboratory. During these years we further improved the calorimeter to make it a more versatile and easily operable research tool.

All these improvements are incorporated in the ADL Model-6 Calorimeter supplied to you.

The prototype calorimeter has been used for measurement of the thermal conductivities of powders such as perlite, colloidal silica, olivine, granodiorite, and tektite; vesicular materials such as sintered perlite, pumice and basalt; and solid rocks such as granite. The operation of the apparatus and the results obtained in the temperature region from 77 to 400°K at gas pressures from 10^{-6} torr to atmospheric are discussed in the Summary Reports under Contract No. NAS8-1567.[2,3] The guarded cold plate apparatus has also been used for measurements of the emittance of plastic, metallic, and non-metallic materials and for the measurement of the heat flux through layers of solid and powdered materials under mechanical compression from 0 to 20 psi. The cold plate apparatus will be useful in determining the thermal properties of simulated lunar materials, materials characteristic in other planetary environments, thermal insulations, and other non-metallic materials.

In addition to the improvements which result from years of operation of the 6-inch diameter calorimeter, several of the design features of the more advanced ADL Model-12 Calorimeter, which was conceived and manufactured by Arthur D. Little, Inc., approximately three years ago,[4] are incorporated in the Model-6 Calorimeter. These features are:

1. Capacity of the guard vessel is enlarged by 38% to increase the time between fillings, thereby providing for longer uninterrupted tests.

2. Copper temperature equalizers are incorporated in both guard and measuring vessels, thereby eliminating a possible error due to stratification of the liquid.
3. Measuring vessel is supported by three 1/4-inch O.D. tubes rather than one 3/8-inch O.D. tube. Three tubes impart increased rigidity to the vessel assembly, permit more precise measurement of the pressure in the measuring vessel, and provide a built-in fill line which simplifies the filling operation.
4. All six lines (three to the measuring vessel and three to the guard vessel) pass through the same opening in the bell jar rather than through two openings. Assembly of the apparatus is thereby simplified.
5. The space between the guard and measuring vessels is connected by a 1/2-inch diameter opening to the bell jar, thereby permitting easy evacuation of that space.
6. A step is provided in the gap between the measuring and guard vessels. This step reduces the possibility of stray radiation reaching the sides of the measuring vessel.
7. The sample chamber is made from thin, 300 series stainless steel. This chamber, together with a flange on the guard vessel, assures that the stainless steel diaphragm covering the sample chamber will be closer to the temperature of the cryogen contained in the guard vessel.
8. The O-ring type seal which was successfully used in the Model-12 Calorimeter is used to isolate the vacuum in the bell jar from the atmosphere in the sample chamber.
9. The stationary warm plate-moving cold plate arrangement used in the original unit has been replaced by the stationary cold plate-moving warm plate system designed for the Model-12 Calorimeter.

10. Diameters of the passages for pumping the bell jar and the sample chamber are increased from 2 to 4 inches and from 3/4 to 2 inches, respectively. In addition, all vacuum connections have been relocated to the base plate of the Calorimeter. The new location simplifies the changing of the test sample.
11. The system which was successfully used on the Model-12 Calorimeter for vertical positioning of the warm plate and for applying compression to the sample is utilized in the new Model-6 Calorimeter.
12. A new spiral arrangement of the coil inside the warm plate was designed. The new coil provides more uniform heating of the warm plate.
13. A detachable plate carrying four thermocouples was added to the warm plate. It facilitates easy replacement, if necessary, of the fine thermocouple wires.
14. The thermocouple feedthrough used successfully on the Model-12 Calorimeter was adopted for the Model-6 unit.
15. An arrangement for lifting and guiding the bell jar and the cold plate has been added.
16. The size of the bell jar has been increased to accommodate a larger guard vessel and the new arrangement of the vacuum connections.

1.2 CAPABILITIES OF THE CALORIMETER

The ADL Model-6 Calorimeter is designed to measure thermal conductivity of solid, powder, and fibrous materials. The effects of variables such as the type of gas penetrating the sample, gas pressure, compressive loading, boundary temperatures, density, and sample thickness can be studied in the calorimeter.

The test sample in the form of a disk can range in thickness from 0 to 1 inch with diameters up to 6-3/4 inches; the measured section of the sample is 3-7/16 inches in diameter. A known compression force ranging from 0 to 20 psi can be exerted on the sample without interruption of the test by application of a mechanical load through an externally

operated hydraulic jack. The environment of the sample can be varied from 1 x 10^{-5} torr to 10 psia for any desired gas, without interruption of the test. One side of the sample can be exposed to a range of discrete temperatures from 77°K to 270°K, which can be achieved by the use of various liquids boiling at low temperatures. The other side of the sample may be exposed to a range of temperatures from 77°K to 450°K by proper choice of fluids. The thermostatically controlled oil bath supplied with the calorimeter can control the temperature of the heat-source plate only between room temperature and 400°K.

1.3 DESCRIPTION OF THE CALORIMETER

Figure 1 shows a general view of the ADL Model-6 Calorimeter. Figure 2 presents a vertical cross section through the calorimeter shells.

The ADL Model-6 Calorimeter consists of the following components:

1.3.1 Measuring and Guard Vessels (Cold Plate Assembly)

The 300 series stainless steel inert-gas-welded cold plate assembly shown in Figure 3 consists of a 3-1/4-inch O.D. by 6-inch high measuring vessel (0.66-liter capacity) enclosed by a ring-shaped guard vessel (7.2-liter capacity) made of an 8-inch O.D. by 15-inch high outer shell and 3-1/2-inch O.D. by 12-inch high inner shell. A 1/16-inch wide gap separates the O.D. of the measuring vessel from the I.D. of the guard vessel. This gap, when evacuated, decreases the heat transfer from vessel to vessel. The measuring vessel is provided with three 1/4-inch O.D. by 0.020-inch thick wall, 300 series stainless steel tubes, which serve as vent, relief and fill lines. All three tubes run for 6 inches inside an evacuated space between the top of the measuring vessel and the guard vessel to reduce possible heat leaks along these lines in case the temperatures of the measuring and guard vessels should differ. These tubes pass through the guard vessel where they are pre-cooled to the temperature of the cryogen contained in the guard vessel. This design feature, and the enclosure of the measuring vessel on all sides but the bottom by the guard vessel, thermally isolates the measuring vessel from any direct contact with the ambient temperature.

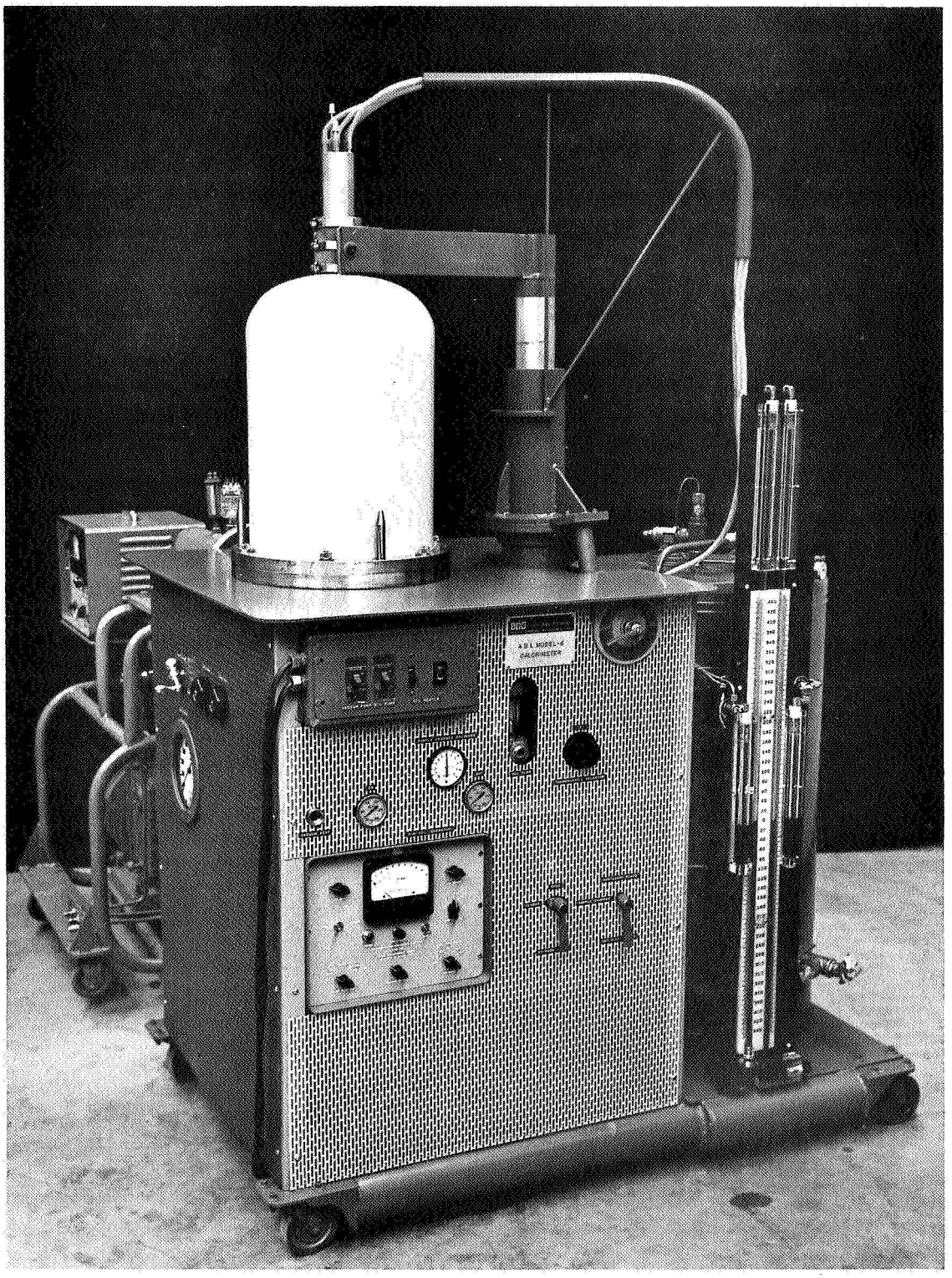


FIGURE 1 GENERAL VIEW OF THE ADL MODEL-6 CALORIMETER

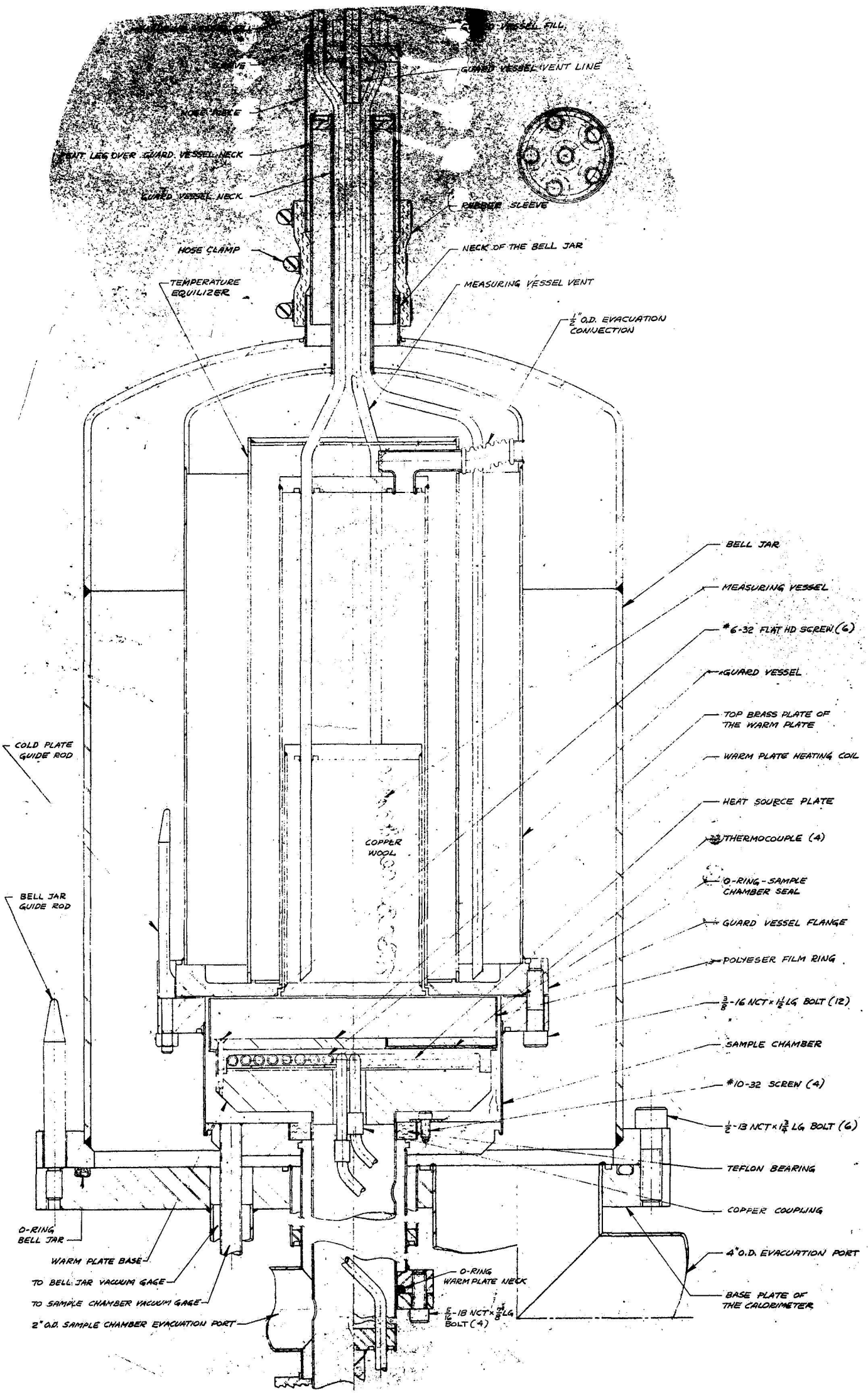


FIGURE 2 ADL MODEL-6 CALORIMETER
(Cross Section)

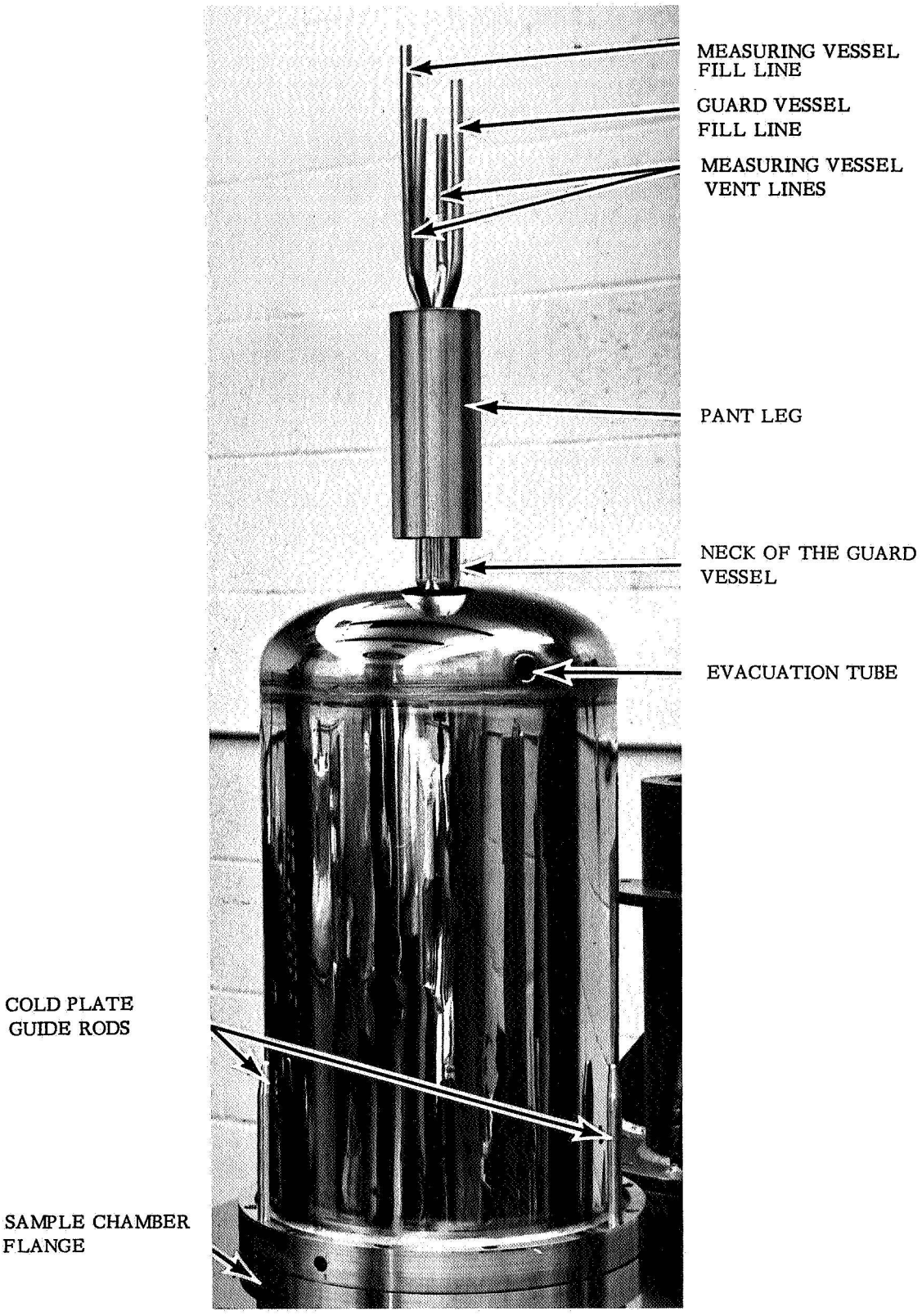


FIGURE 3 MEASURING AND GUARD VESSELS (COLD PLATE ASSEMBLY)

All three lines from the measuring vessel, as well as the 1/4-inch O.D. guard vessel fill line, pass through the 1-inch O.D. guard vessel neck, which simultaneously serves as a vent for the guard vessel. The neck of the guard vessel is 6 inches long and is provided with a 2-inch O.D. pant leg in order to reduce the heat conducted from the ambient temperature to the guard vessel along the neck. During normal operation, the space between the neck and the pant leg is evacuated, decreasing heat conduction from the pant leg, which is exposed to ambient temperature, to the neck. These two design features decrease the boil-off rate of the liquid inside the guard vessel, thereby increasing the length of operation of the guard vessel with the same charge of cryogen.

The neck of the guard vessel passes through the 2-1/4-inch O.D. neck at the top of the enclosing bell jar (see Section 1.3.4 below). A 2-inch I.D. by 3-inch long rubber sleeve covers the gap between the neck of the bell jar and the pant leg of the guard vessel's neck. The space between the pant leg and the neck of the guard vessel forms part of the bell jar vacuum space. Two hose clamps seal the rubber sleeve seal.

The same rubber sleeve provides a tight joint between the nose piece (see Figure 2) and the pant leg. The nose piece seals the inside of the guard vessel, permitting control of the pressure of the cryogen in the guard vessel. The three lines from the measuring vessel and guard vessel fill line pass through 5/16-inch O.D. by 5/8-inch long sleeves protruding from the top of the neck piece. The gaps between the lines and the sleeves are sealed with 1/4-inch O.D. by 1-inch long pieces of a gum-rubber hose. The nose piece has two more sleeves: one, which begins at the top of the nose piece, is used for measuring the pressure in the guard vessel (see Section 1.4.5); the other, which protrudes 1 inch below the top of the nose piece, serves as a vent line for the guard vessel. Since the venting gas flows through a line below the pressure indicating tap, there is no flow at the opening of the pressure measuring line; therefore, it is possible to measure a static gas pressure in the guard vessel.

A 6-1/2-inch O.D. by 1/16-inch wall thickness by 13-inch high copper temperature equalizer is placed inside the guard vessel. The equalizer

serves to eliminate stratification of the cryogen in the guard vessel and to conduct heat away from the vent lines of the measuring vessel when the liquid level in the guard vessel is low. Copper wool is placed inside the measuring vessel to eliminate stratification of the cryogen.

The cylindrical and the top surfaces of the measuring and guard vessels facing the vacuum space are nickel-plated to provide low emittance surfaces to reduce the radiative heat transfer to the vessels. The space between the guard and measuring vessels is connected to the rest of the bell jar by a 1/2-inch diameter pipe tee. This connection permits evacuation of that space, particularly when a stainless steel diaphragm is used over the sample chamber (see Section 1.3.3). The bottom plate of the measuring vessel is 3-7/16-inch in diameter (11.8 square inches in area) by 5/16-inch thick. The bottom flange of the guard vessel is 9-1/4-inch diameter. The 7-inch diameter portion, which is facing the inside of the sample chamber, is also 5/16-inch thick. This assures that the temperature drop through the thickness of the guard vessel flange is the same as for the measuring vessel flange. For powder or foam insulation, the error made by assuming zero temperature rise through the flange is below 1%.

The outer portion of the guard vessel flange is 7/8-inch thick. It bolts to the mating flange of the sample chamber. The heavy outer portion of the flange provides the stiffness necessary to maintain a a cold seal between the mating flanges.

The 7-inch diameter portion of the guard vessel flange, as well as the bottom surface of the measuring vessel, constitute the guarded cold plate and, simultaneously, the top of the sample chamber. The cold plate is covered with a black paint of 0.86 emissivity to provide a surface of known emittance for determining the radiation portion of the heat transfer through a sample during the test.

At the bottom of the measuring vessel flange, a 1/16 by 1/16-inch step is provided in the gap between the measuring and guard vessels. The step reduces the possibility of stray radiation reaching the sides of the measuring vessel.

Three equally-spaced holes on the circumference of the guard vessel flange are provided for guide rods (see Section 1.3.3). The guide rods prevent dislocation of the test sample when the cold plate assembly is lowered over the sample during assembly of the equipment in preparation for a test.

1.3.2 The Warm Plate Assembly (Figure 4)

The horizontally placed warm plate supports the sample and consists of a 6-3/4-inch diameter by 1/4-inch thick brass plate, covered on the top side by a black paint of 0.86 emissivity. Four #36 (.005" diameter) copper-constantan thermocouples are embedded flush with the upper surface of the plate. The thermocouple leads are cemented with copper cement in the grooves milled in the lower surface of the same plate. Figure 5 shows the location of these thermocouples.

The plate is placed over a 6-1/2-inch diameter brass heat source plate in which a spiraled copper coil is cemented (Figure 6). The coil serves to maintain the warm plate at a desired constant temperature by means of fluid flowing through its passages. The fluid temperature is regulated in the constant temperature bath described in Section 1.4.3 below. The heat source plate, together with the upper 1/4-inch thick brass plate is bolted with 6 flat-headed brass screws to the base of the warm plate assembly. The base is a 6-1/2-inch diameter by 1-inch thick brass disk. It provides the desired stiffness for the warm plate assembly. The base is supported by the warm plate neck. The 2-inch O.D. x 0.28-inch thick wall x 13-5/8-inch long neck is made from 300 series stainless steel. The long neck protects the heat source plate from an excessive heat exchange with the ambient temperature. When the sample chamber is evacuated, the inside of the neck is evacuated also, providing a better thermal insulation. For this purpose, the neck has four equally spaced 1/2-inch diameter holes 1-7/8 inches below the top of the neck. Another set of identical holes is located approximately 3-3/4 inches above the lower end of the neck.

The two 1/4-inch O.D. stainless steel fluid supply lines run inside the neck to the heat source plate. The lines are slightly spiraled to

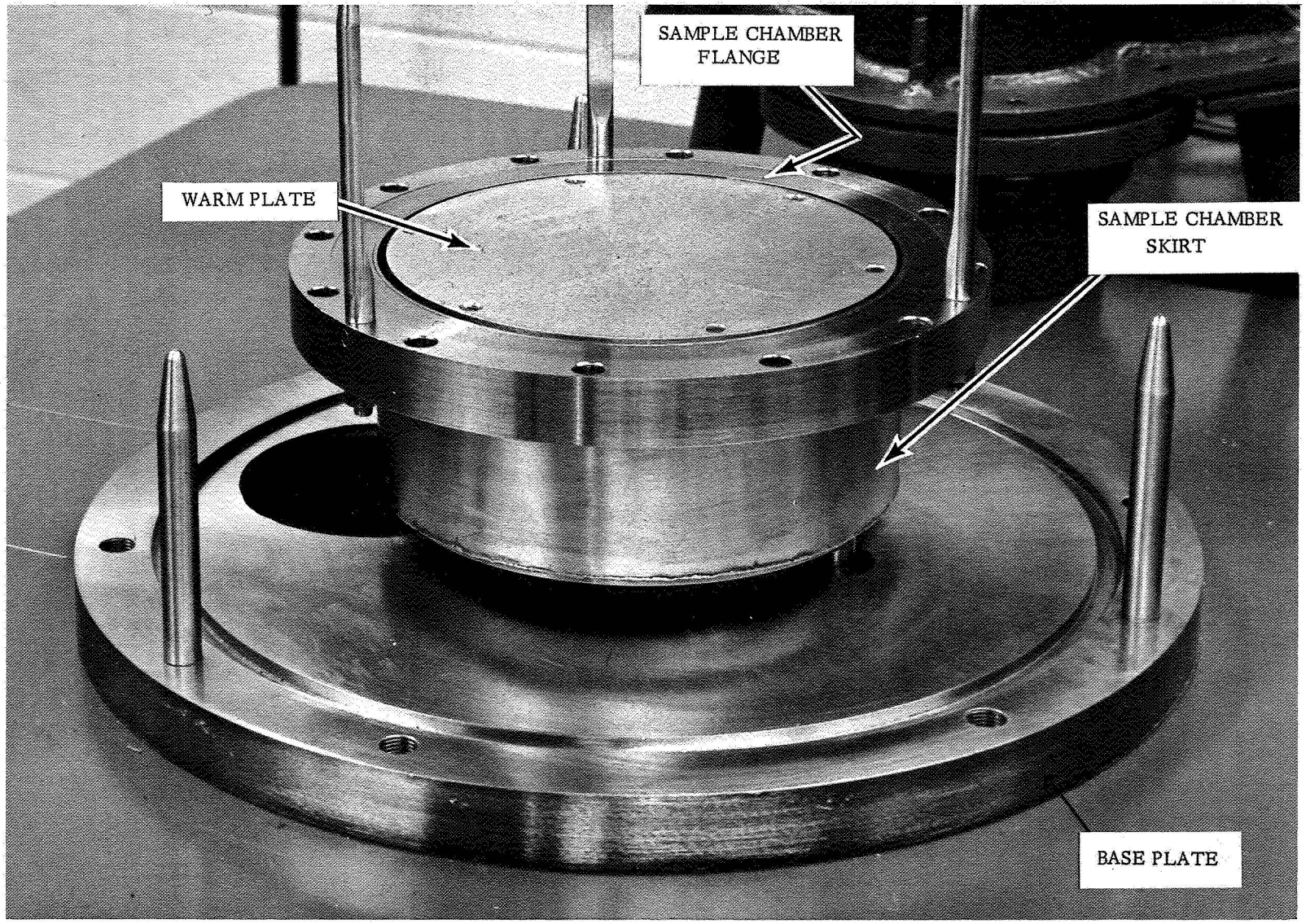


FIGURE 4 WARM PLATE, SAMPLE CHAMBER AND BASE PLATE

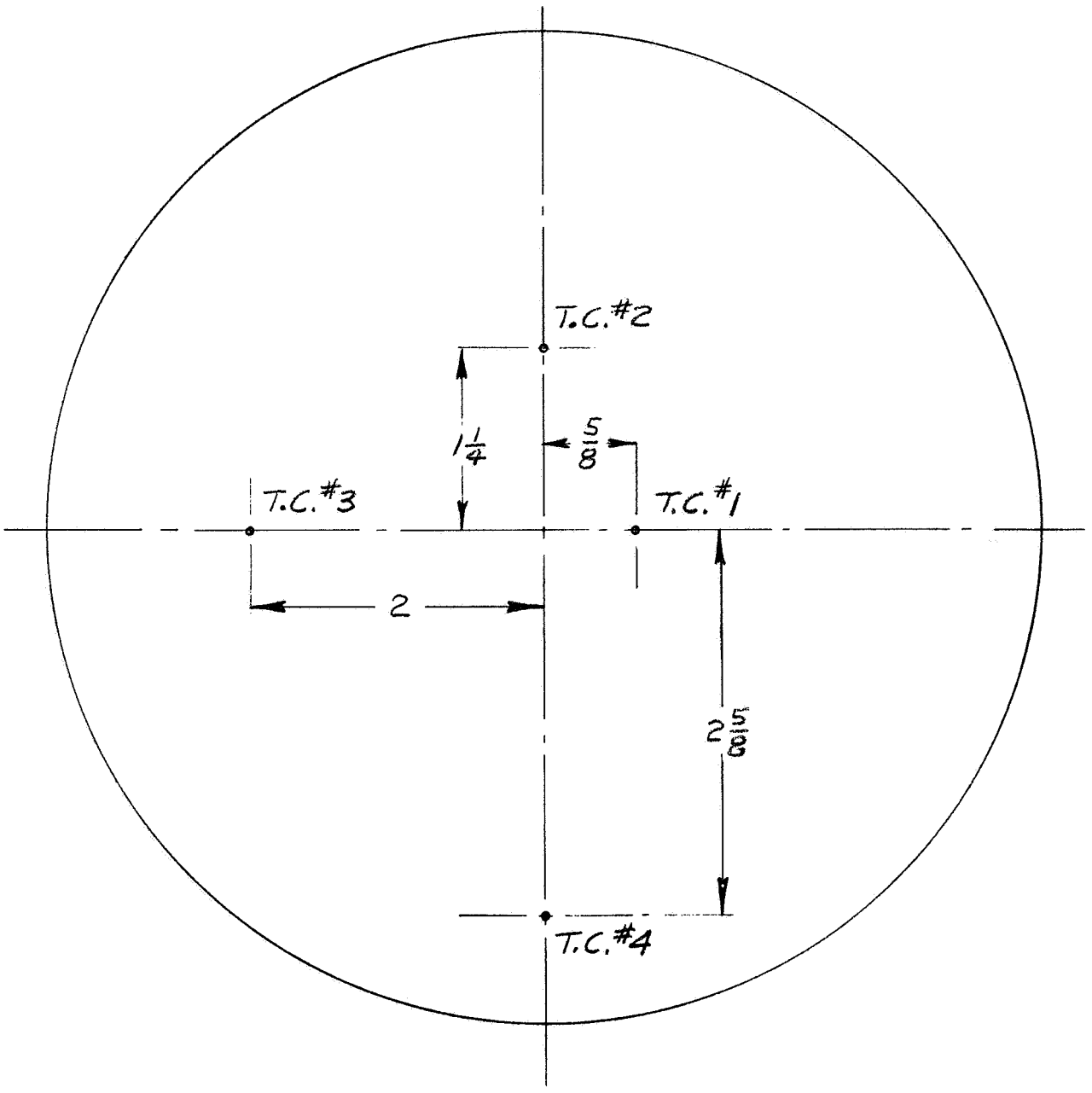


FIGURE 5 LOCATION OF THE THERMOCOUPLES IN THE WARM PLATE

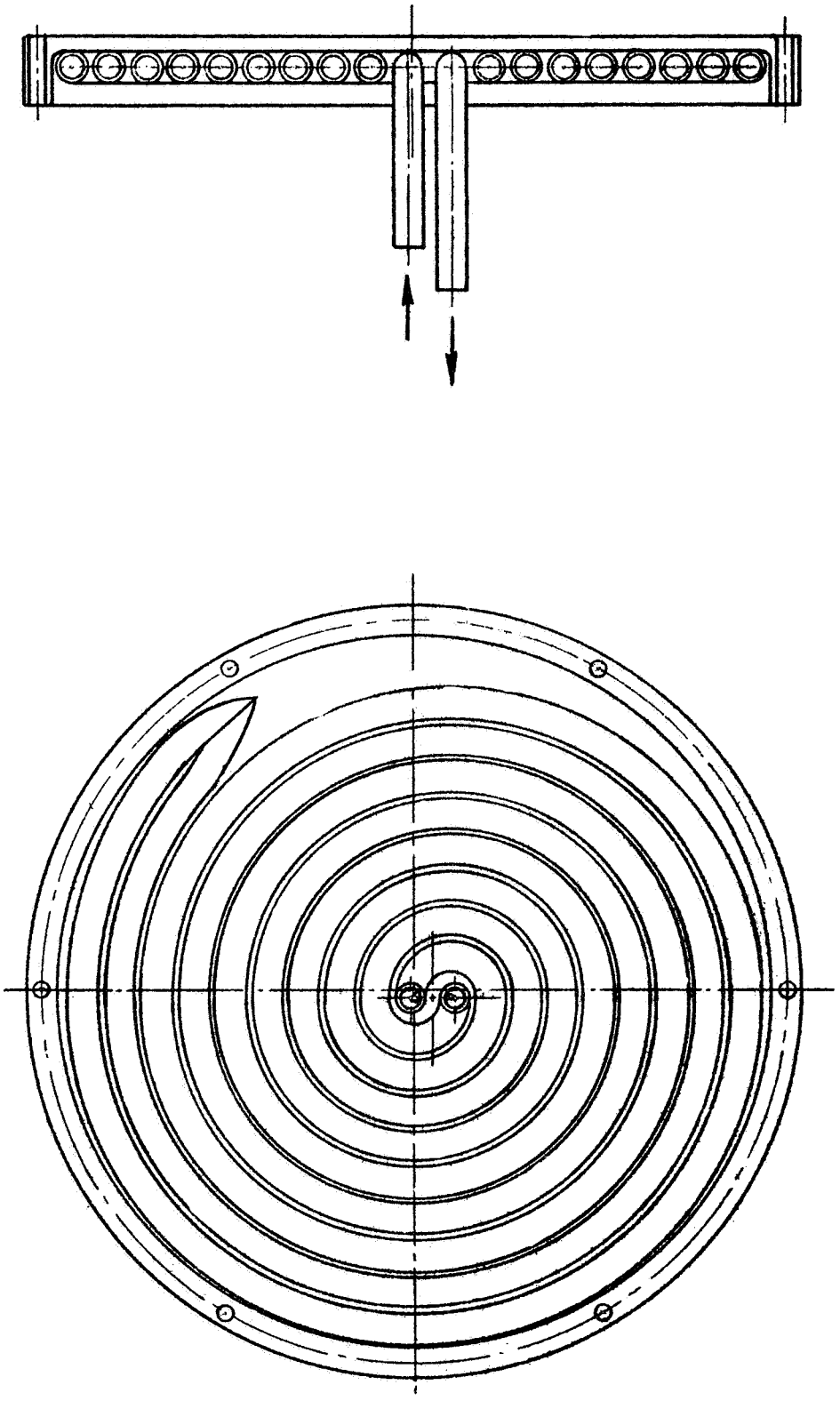


FIGURE 6 WARM PLATE HEATING COIL

provide for thermal expansion. These lines and the two ends of the copper coil protruding from the heat source plate are soldered just below the heat source plate into two copper couplings.

During the adjustment of the vertical position of the warm plate, the neck of the warm plate assembly is guided by a Teflon bearing (Figure 7) (Section 1.3.3) and an 0-ring spaced 11-1/2 inches apart, providing smooth bearing surfaces and isolation of the warm plate from the base plate of the apparatus. The adjustment in the warm plate position is made with the help of a hydraulic jack external to the sample chamber (see Section 1.4.6).

A ring made from 5 mil thick by 1-1/4-inch wide polyester film fits over the 6-3/4-inch O.D. of the top brass plate of the warm plate. The use of the ring is necessary only at times when powdered materials are under test; it prevents the powder from flowing over the edges of the warm plate. The ring can slide down along the edge of the warm plate and, therefore, does not interfere with the raising of the warm plate during a test.

1.3.3 Sample Chamber (Figures 4 and 8)

The sample chamber is bounded by the guarded cold plate on the top, by the 3/4-inch thick base at the bottom and by the 7-inch I.D. x 3-inch high skirt at the sides. A 9-1/4-inch diameter by 7/8-inch thick flange is welded at the top of the sample chamber skirt. The base carries a 1/2-inch diameter connection for measuring vacuum (or pressure) in the sample chamber. The sample chamber is supported from the base plate of the calorimeter by a 2-1/2-inch O.D., 0.020-inch wall neck welded to the base with a 3-inch O.D. by 0.028-inch thick wall by 7-inch long pant leg around it. The vacuum between the neck and its pant leg is common with the bell jar vacuum. This vacuum and the length of the neck thermally isolate the sample chamber from the ambient temperature. For the same purpose, the base of the sample chamber is raised 1/4-inch above the base plate of the apparatus (see Section 1.3.5 below). The whole assembly is made from 300 series stainless steel. At the bottom, the sample chamber neck is sealed from the ambient conditions by an arrangement of two flanges and a 2-inch I.D. by 3/16-inch cross section 0-ring. The neck of the warm

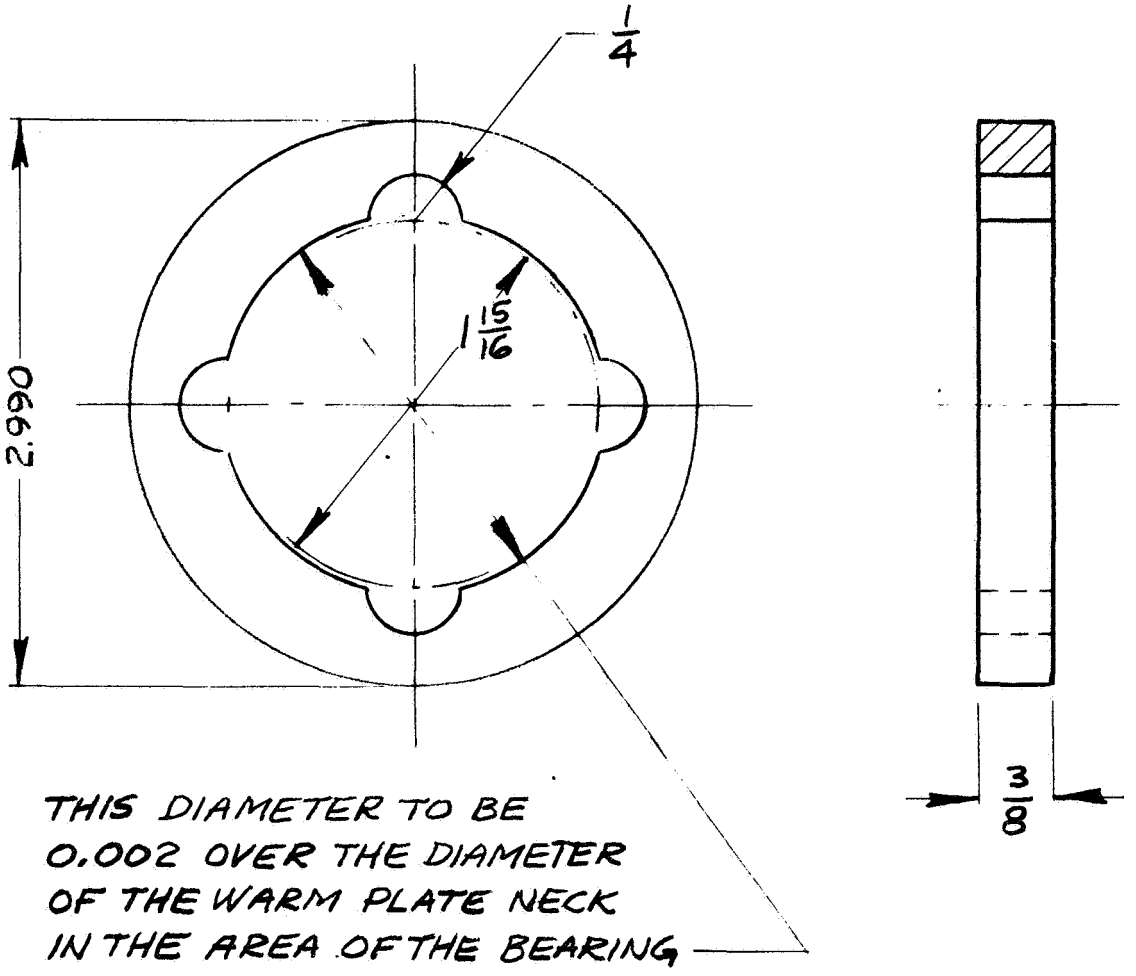


FIGURE 7 TEFLON BEARING ADL MODEL-6 CALORIMETER

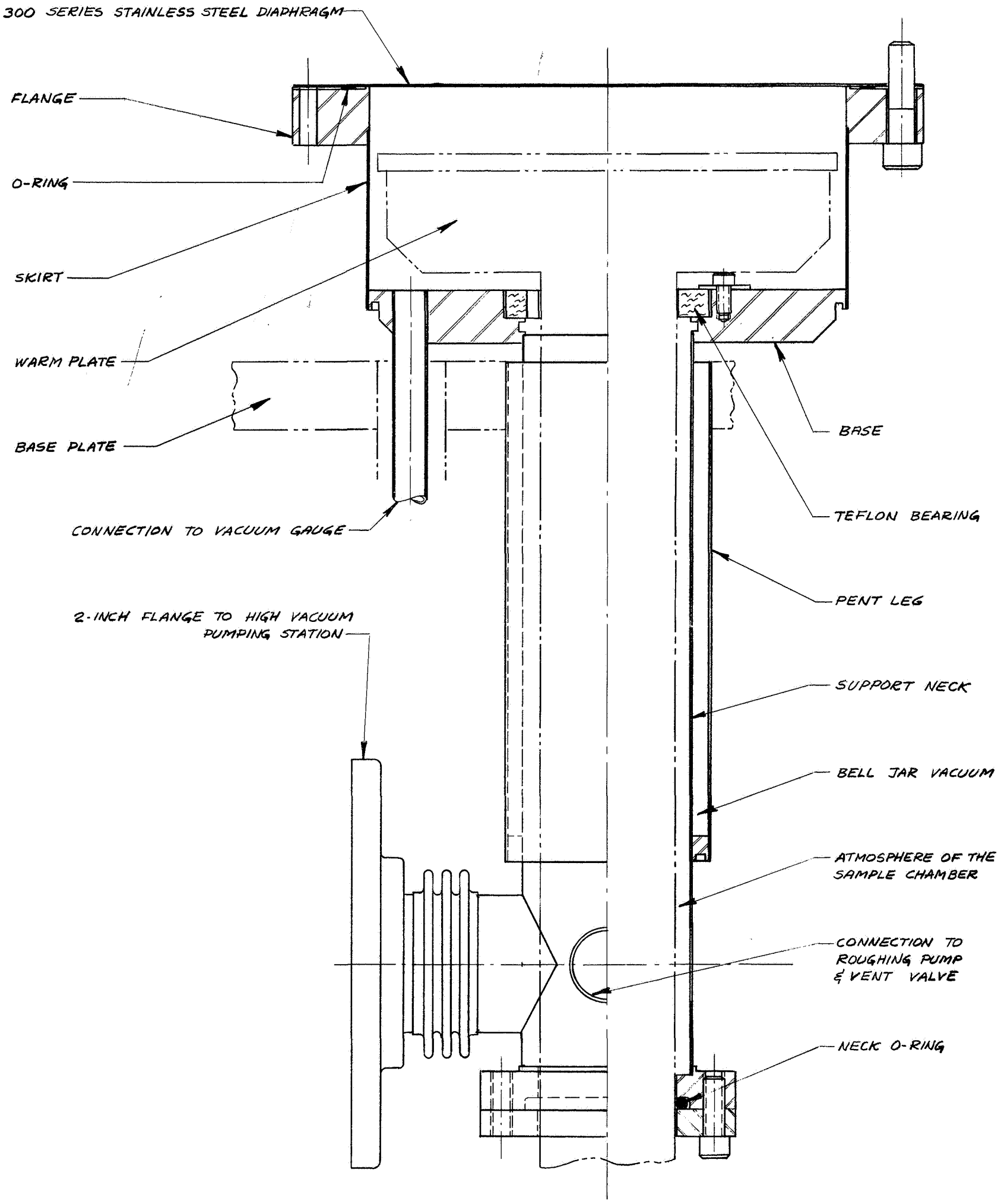


FIGURE 8 SAMPLE CHAMBER

plate (see Section 1.3.2 above) slides through this O-ring and a 3-inch O.D. by 2-inch I.D. x 3/8-inch thick Teflon bearing when the warm plate vertical position is being adjusted.

Just above this seal, the neck of the sample chamber carries one 1-1/8-inch O.D. and one 2-inch O.D. tubing. The 1-1/8-inch tubing is provided with a tee and two manually-operated 1-inch brass block vacuum valves. A 5CFM roughing pump is connected to one of the valves, while the other valve can be used to bleed air or other gases into the sample chamber.

The 2-inch O.D. tubing through a manually-operated, 2-inch gate vacuum valve is connected to the 4-inch O.D. tubing leading from the bell jar to the high-vacuum system (Section 1.4.1). Through various settings of these valves (provided the sample chamber is hermetically sealed from the vacuum in the bell jar) it is possible to evacuate the sample under test to any degree of vacuum or to introduce various gases into the sample chamber. The ring-shaped space between the neck of the warm plate and the neck of the sample chamber serves as a passage between the sample chamber and the service connections described above. The Teflon bearing described above is scalloped on the inner diameter in order to provide passages for evacuation.

A 9-1/4-inch diameter 0.0015-inch thick 300 series stainless steel diaphragm clamped between the top flange of the sample chamber and the flange on the guard vessel (Section 1.3.1) is used to hermetically seal the sample chamber from the surrounding vacuum space of the bell jar. A highly compressed 7-1/2-inch I.D. by 1/16-inch cross section, neoprene O-ring provides the seal between the diaphragm and the flange of the sample chamber.

1.3.4 Bell Jar (Figure 9)

The warm plate, the cold plate assembly, and the sample chamber are enclosed in a 12-3/4-inch O.D. by 19-inch high, 300 series stainless steel bell jar. The bell jar can be evacuated by the high vacuum pumping system described in Section 1.4.1 below. The bell jar is placed on a base plate (see Section 1.3.5 below). An O-ring provides a vacuum-tight seal between

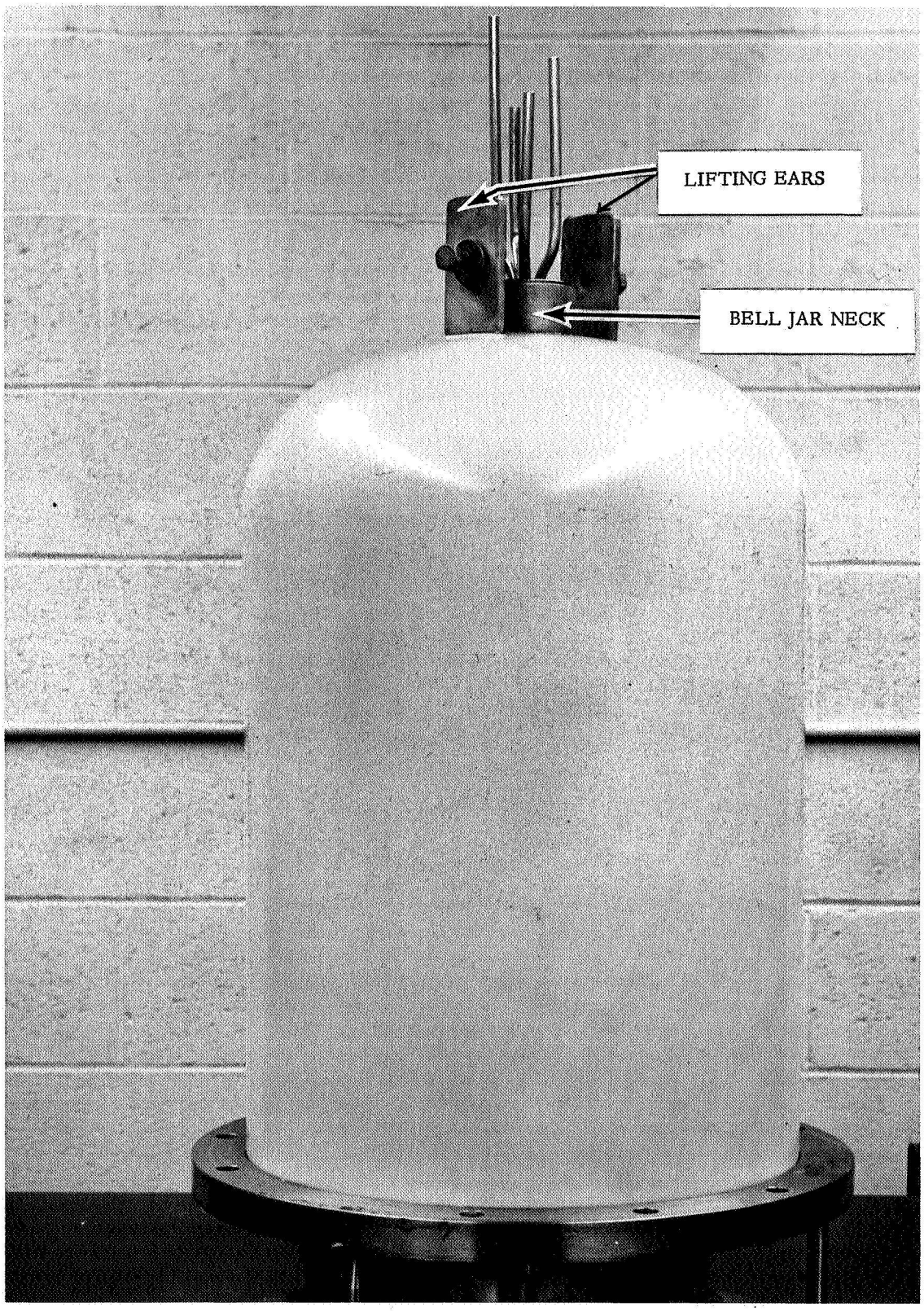


FIGURE 9 BELL JAR

the flange of the bell jar and the base plate. A 2-1/4-inch O.D. by 0.028-inch thick neck protrudes 1 inch above the top of the bell jar. The neck of the guard vessel which carries vent and fill lines for both vessels passes through this neck. A 2-inch I.D. by 1/4-inch thick by 3-inch long sleeve made from natural rubber serves to seal the gap between the neck of the bell jar and the neck of the guard vessel. The bottom flange of the bell jar has 6 equally-spaced clearance holes for 1/2-inch diameter bolts. These bolts provide initial compression for the 0-ring between the flange and the base plate (when the vacuum in the bell jar is established, atmospheric pressure provides sufficient compression of the 0-ring for it to seal).

In addition to the bolt clearance holes, three other 9/16-inch diameter, equally-spaced holes are provided in the flange. Guide rods from the base plate are sliding in these holes when the bell jar is lifted or lowered. This arrangement prevents the bell jar from swinging and damaging 0-ring or other components.

Two lifting ears are welded at the top of the bell jar. The ears are provided with 1/2 x 13 N.C.T. bolts connecting them with the fork on the hoist (Section 1.4.7).

1.3.5 Base Plate

Figure 10 shows the drawing of the base plate assembly. The base plate is made from 15-inch diameter by 1-inch thick 300 series stainless steel. In its center it has a 3-inch diameter hole inside which a pant leg of the sample chamber neck is welded (Section 1.3.3). In addition, the base plate has a 4-inch diameter tubing with a flange to which the pumping system described in Section 1.4.1 below can be connected. Another 1-inch opening in the base plate serves for gauging vacuum in the bell jar and in the sample chamber. Two sets of ionization and thermocouple-vacuum gauges are provided for this purpose. The base plate is provided with a 12-1/2-inch I.D. groove for a 1/4-inch cross section 0-ring. The 0-ring, as mentioned in Section 1.3.4, seals the vacuum in the bell jar. Three equally-spaced 1/2-inch diameter by 4-inch long guide rods are bolted into the base plate (see Section 1.3.4).

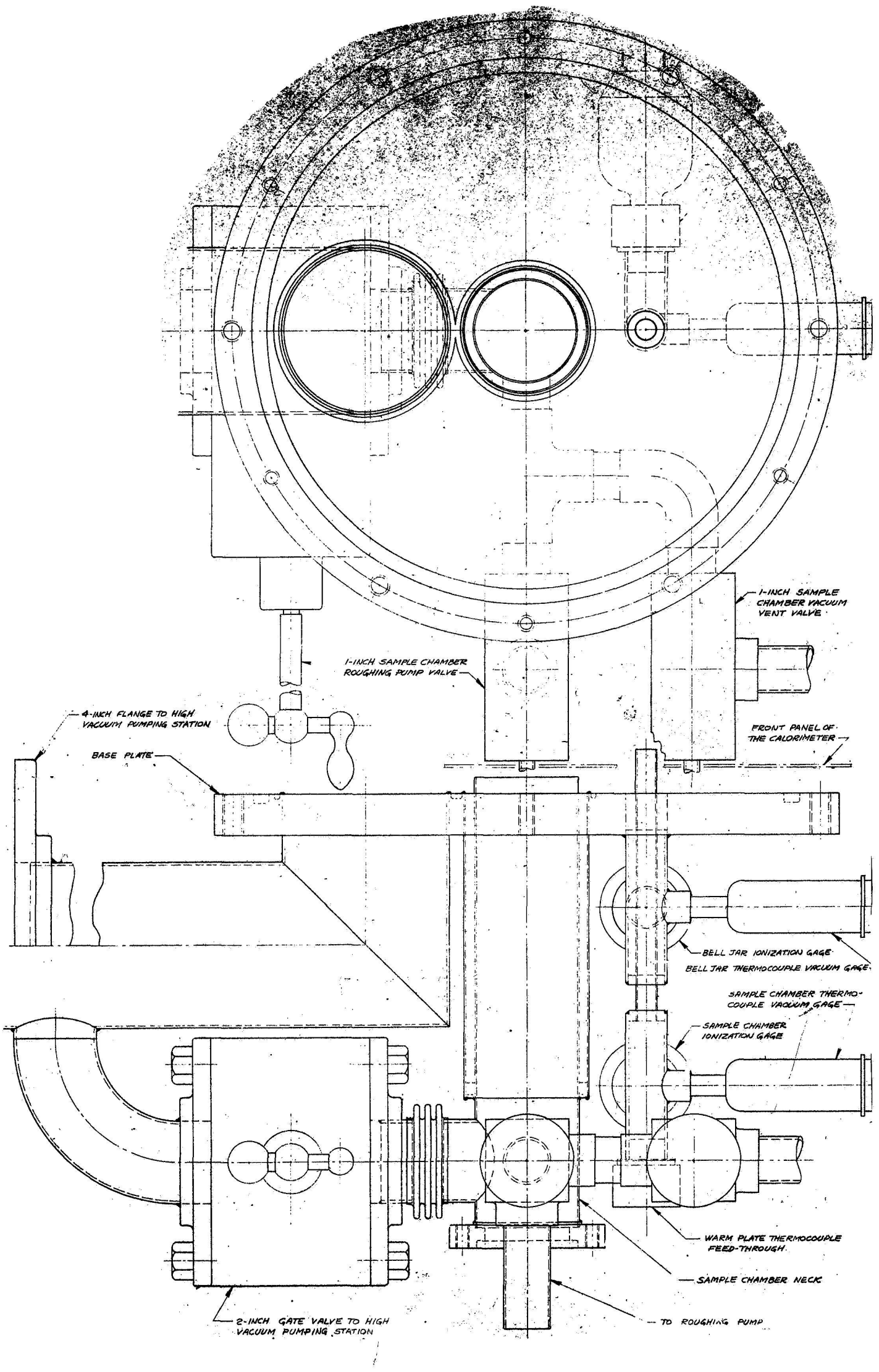


FIGURE 10 BASE PLATE ASSEMBLY
ADL-MODEL-6 CALORIMETER

1.4 INSTRUMENTATION

Figure 11 shows the schematic of the instrumentation necessary for the operation of the ADL Model-6 Calorimeter. Instrumentation shown by dotted lines is not supplied by Arthur D. Little, Inc.

1.4.1 Vacuum Pumping System

The vacuum pumping system supplied with the calorimeter is presented schematically in Figure 12. The system consists of a 4-inch detachable high-vacuum pumping station (NRC, Model 3305), a 140 liter/min roughing pump (Welch, Model 1402B) and a system of vacuum valves.

The 4-inch, high-vacuum pumping station is a self-contained unit which is connected to the 4-inch O.D. tube flange of the base plate (Section 1.3.5). The station is described in the supplied NRC Manual. During the test, this vacuum station evacuates and maintains high vacuum (below 1×10^{-5} torr) in the bell jar. In addition, it evacuates and maintains vacuum in the sample chamber (Section 1.3.3) when a vacuum above 1×10^{-2} torr is required.

Primary function of the Welch roughing pump is to evacuate and maintain vacuum in the sample chamber when a vacuum below 1×10^{-2} torr is required. However, by manipulation of the valves, it can be used for roughing of the bell jar and sample chamber before the test. This feature permits the operator to keep the high-vacuum pumping station clean and ready operating behind the closed 4-inch gate valve while the test sample is being changed.

1.4.2 Measurement of the Vacuum in the Bell Jar and Sample Chamber

As was mentioned in Section 1.3.5, the base plate of the calorimeter is equipped with two ionization gauges (NRC, Model 518) and two thermocouple-vacuum gauges (NRC, Model 501). (See Figures 10 and 12.) The upper station consisting of one thermocouple-vacuum gauge and one ionization gauge serves to measure vacuum in the bell jar. The ionization gauge control supplied with the NRC high-vacuum pumping station (see Section 1.4.1) serves as the read-out instrument for this pair of gauges.

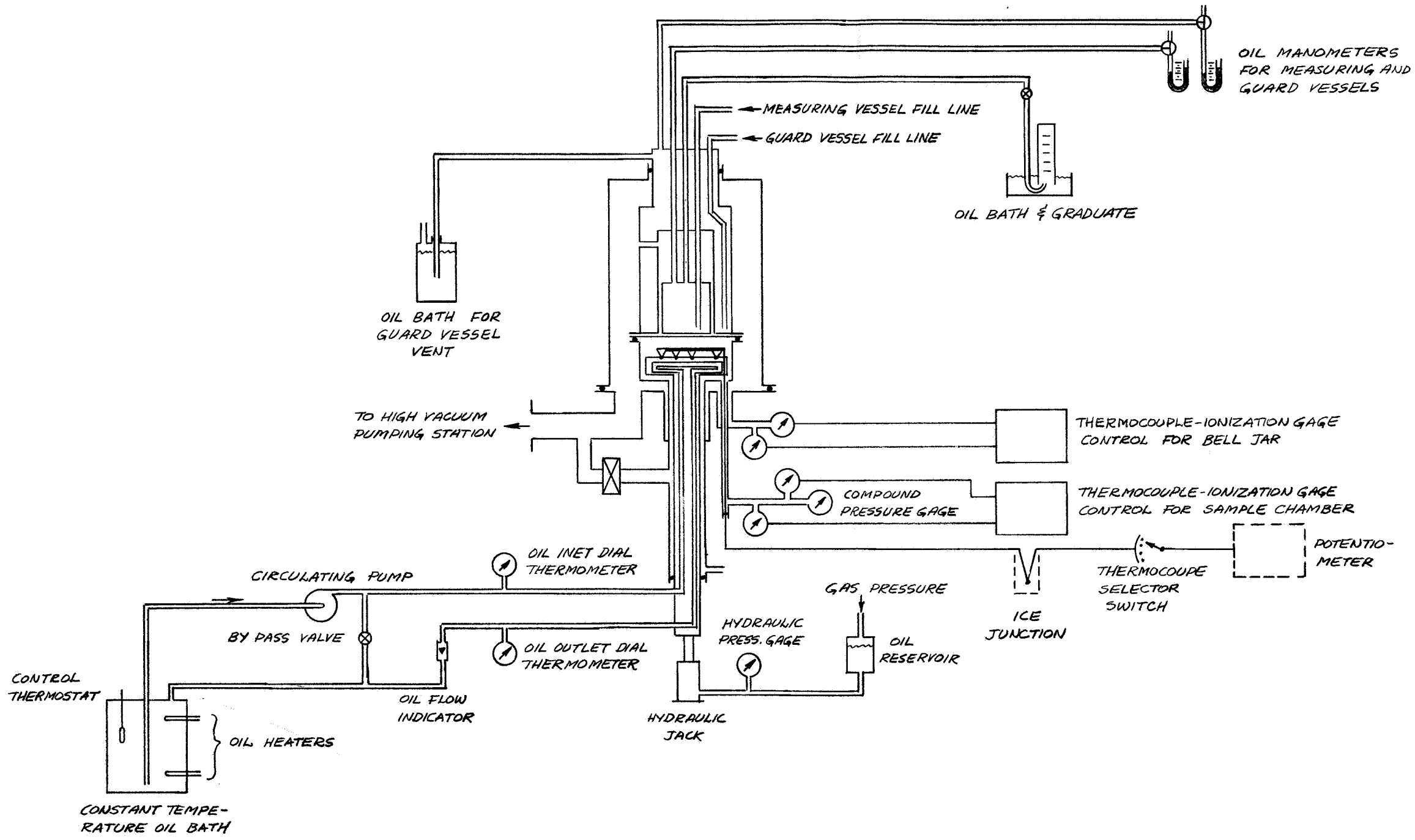


FIGURE 11 INSTRUMENTATION

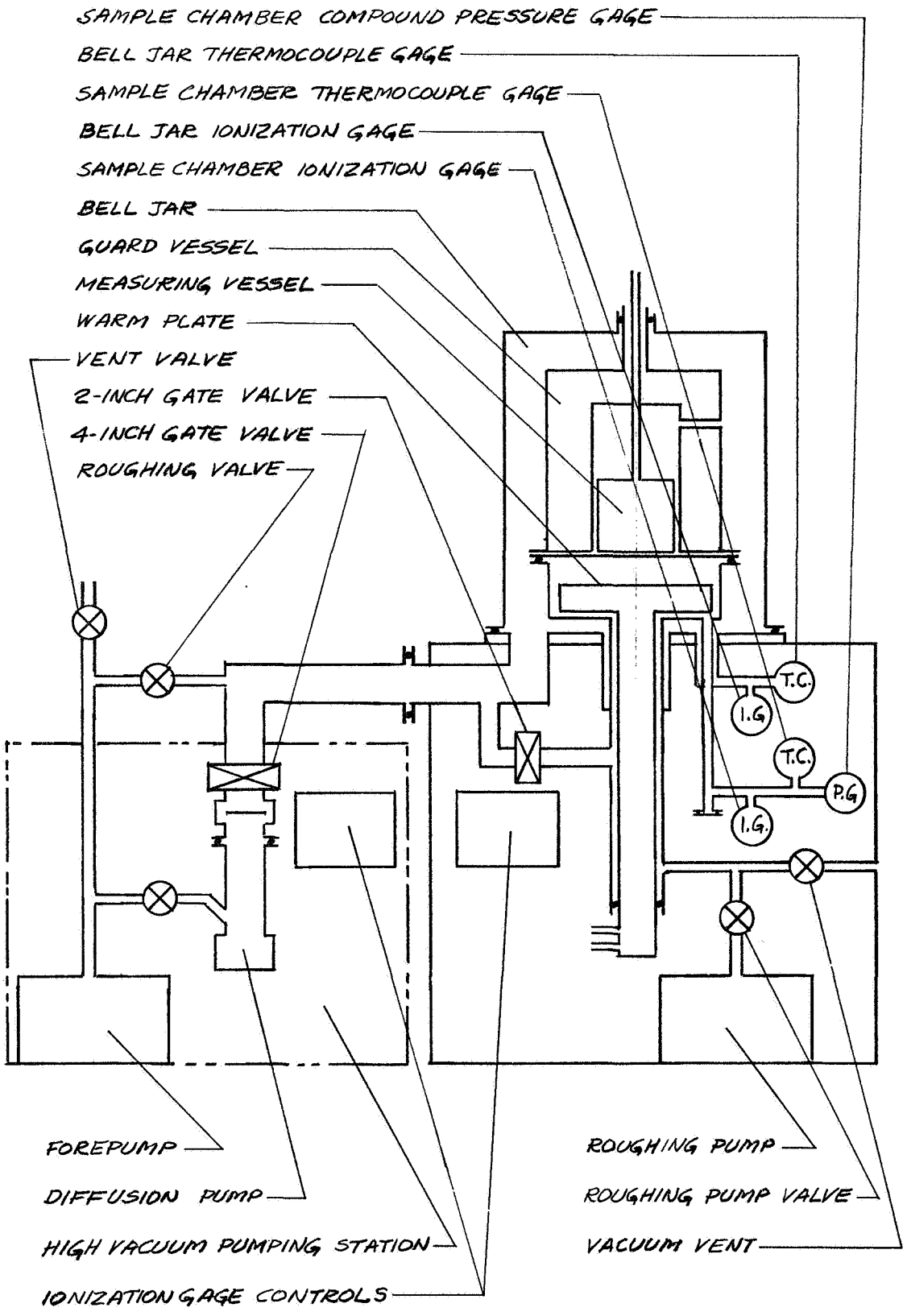


FIGURE 12 VACUUM SYSTEM

The lower station, consisting of two identical gauges, serves to measure vacuum in the sample chamber. In addition, this station has a compound pressure gauge with the range from 30 inches of mercury vacuum to 15 psig pressure (see Figure 13). Ionization and thermocouple-vacuum gauges are read on the ionization gauge control supplied with the calorimeter (Figure 13). Using all three gauges, the pressure in the sample chamber can be read from 1000 torr to 10 torr on the compound gauge, from 1 torr to 5×10^{-3} torr on the thermocouple gauge and from 5×10^{-3} down to 1×10^{-7} torr on the ionization gauge.

During the normal operation of the calorimeter (provided outgassing of the sample is negligible, the vacuum system and the high-vacuum pumping station are clean) the vacuum in the bell jar should be below 5×10^{-5} torr warm and around 1×10^{-6} (or below) when liquid nitrogen is used in the guard and measuring vessels. Sample chamber pressure usually runs approximately a decade above the bell jar pressure (5×10^{-4} torr warm and 1×10^{-5} or below cold).

1.4.3 Measurement and Control of the Warm Plate Temperature

To measure the temperature of the warm plate, four 5-mil diameter copper-constantan thermocouples are provided. (See Section 1.3.2 above.) The thermocouple leads are connected to a selector switch (see Figure 13). The corresponding wires from the selector switch should be connected to an ice junction and to a suitable potentiometer (such as L & N, K-3 type), or a recorder.

To maintain the warm plate at a required temperature, the heating coil of the warm plate is connected to the constant temperature oil bath. The bath permits the warm plate to be kept at any steady temperature between the room temperature and 400°K. The oil bath is a 30-gallon capacity industrial liquid heater (Dayton #3E937). A wide-range precision thermostat (United Electric Control, Type D5) and two heavy-duty heating elements (Chromolox #TG 130, 3000 watts each, used in series) are suitable for heating oil. A 16 GPM capacity turbine type pump (Deming, Model 3900, 3/4" size) circulates the oil (Cities Service, 300T Pacemaker Turbine Oil) through the warm plate and the bath. Two three-way valves are supplied with the circulating system. One--the bypass valve--permits circulation

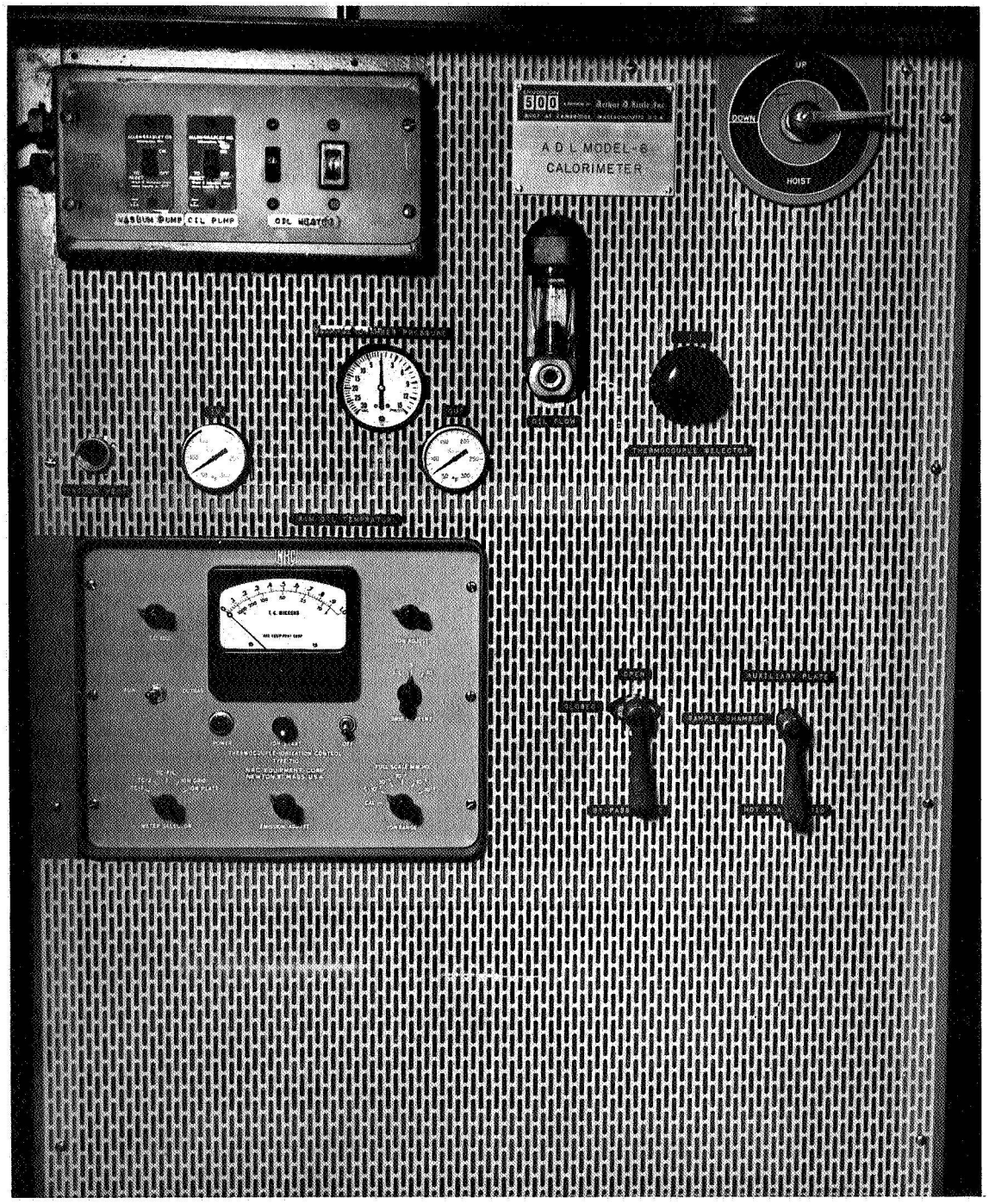


FIGURE 13 RIGHT SIDE CONTROL PANEL

of the oil through the pump and the bath only, thereby permitting to start the warm-up cycle at any time without affecting the temperature of the warm plate. The other valve permits diversion of flow from the warm plate to an auxiliary plate (see Section 2.3.3 below) used with the probe and the line heat source.

When the oil flow is circulated through the warm plate, two dial thermometers (see Figure 13) (Weston, Model 2281) indicate the inlet and outlet temperature of the oil. These thermometers are used only for quick-glance reference of the warm plate temperature. Thermocouples must be used to read the precise temperature of the warm plate. Oil flow indicator installed in the warm plate oil circuit (see Figure 13) shows if the oil flow is normal.

In case the temperature of the warm plate is required to be outside the limits provided by the oil bath capacity, the latter can be disconnected at the flexible tubing near the warm plate. Provided the oil has been carefully washed from the warm plate coil, other cooling or warming fluids or gases can be used. For example, liquid nitrogen flow will provide temperature near 80°K. Refrigeration unit can be attached for temperatures near 270°K. Steam or hot gas can be used above 400°K.

1.4.4 Measurement of Cryogen's Boil-Off Rate

The heat flux passing through the test sample in the calorimeter is calculated from the boil-off rate of the cryogen contained in the measuring vessel (see Section 1.3.1 above). Boil-off rates in excess of 3 liters of gas per hour can be successfully measured with a simple wet test gas meter (such as American Meter Co. AL-17, 0.1 ft^3/rev meter). To measure low boil-off rates (below 3 liters per hour) we supplied with the calorimeter a system consisting of a 2-liter capacity glass graduate placed upside down into an 18-inch wide by 24-inch long by 2-inch deep tray filled with low vapor-pressure oil (Figure 14). The graduate is set with its open end 1/2 inch below the oil surface. Thus, the oil seals the open end of the graduate. The upper (closed) end of the graduate carries 1/4-inch diameter tubing to which a rubber hand pump is attached. The pump is used periodically to suck the oil from

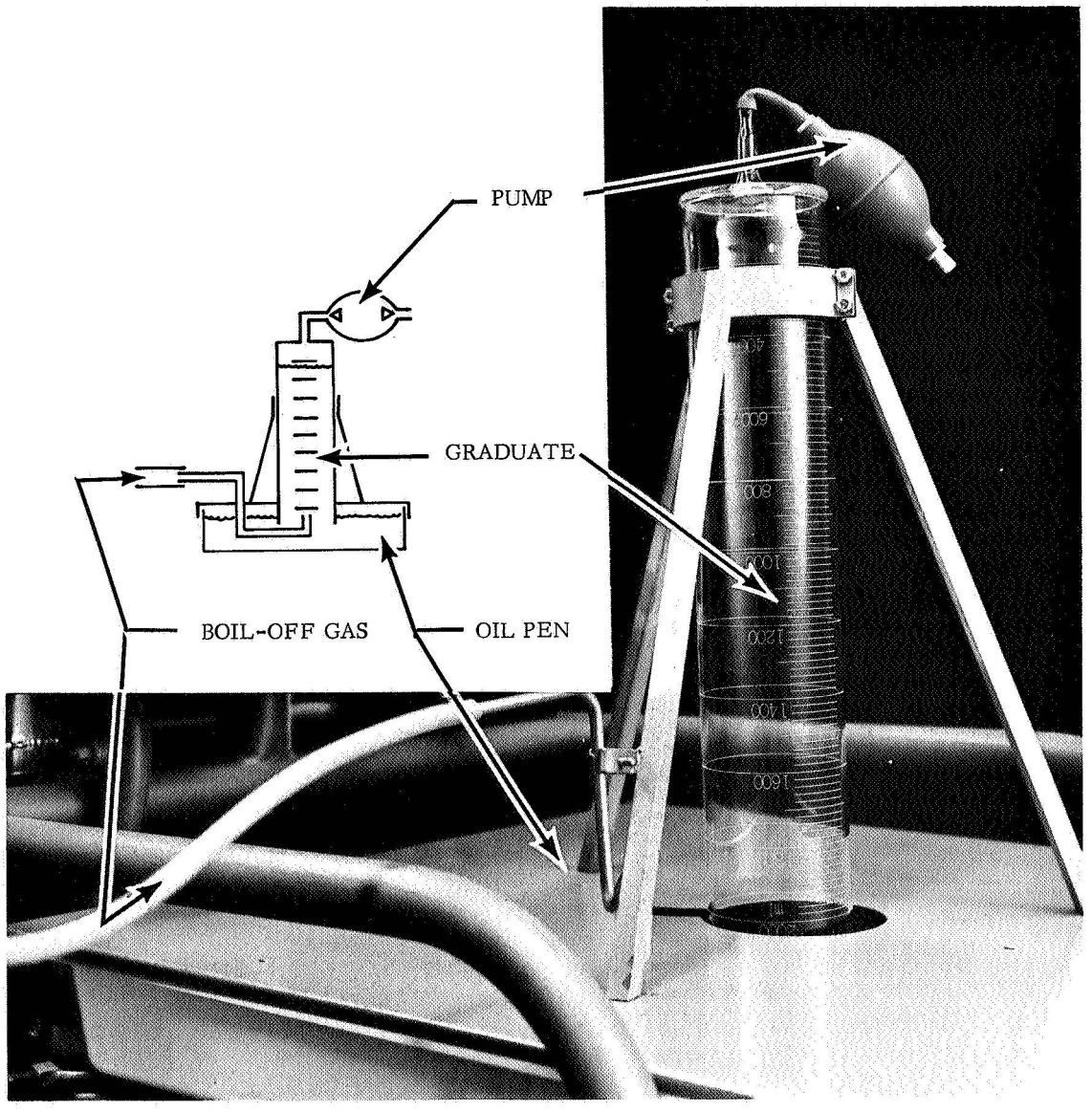


FIGURE 14 BOIL-OFF RATE METER

the tray into the graduate. The large size of the tray is chosen to make a negligible change in the oil level between the time when the graduate is filled with oil from the tray and when the graduate is empty of all the oil in the tray.

One end of a 1/4-inch diameter copper tubing is placed inside the graduate under the oil surface. The other end of this tubing is connected to the vent of the measuring vessel.

During the test, the graduate is periodically filled with oil (using the rubber hand pump); the boil-off gases from the measuring vessel bubble through the oil, displacing it from the graduate. The boil-off rate is calculated from the time interval a known amount of oil is displaced from the graduate.

A sample calculation of the heat flux through a test sample is presented in Section 1.8.2.

1.4.5 Measurement and Control of Cryogen's Pressure

When measuring low heat fluxes, two conditions must be satisfied in order to receive a measurement of a reasonable precision:

1. The pressure of the liquid in the measuring and guard vessels has to be the same. This condition assures that no heat is transferred from vessel to vessel.
2. The absolute pressure of the liquid in both vessels has to stay constant through the duration of the test once the steady state is reached and the measurement is started. This condition assures that the boiling temperature of the cryogen remains constant.

The first condition is easily satisfied by observing the pressure applied to the measuring vessel as indicated on the oil manometer, and by periodically matching the pressure in the guard vessel with this pressure. The pressure in the guard vessel may be varied by changing the immersion depth of the boil-off discharge tube from the guard vessel in the oil bath.

The gauge pressures in both vessels are indicated by the oil manometers (Figure 19) supplied with the calorimeter. The relief line of

each vessel is connected by a 1/4-inch I.D. gum rubber line to the corresponding oil manometer.

To maintain the absolute pressure in both vessels at the constant level is more difficult because of the variation in barometric pressure.

Depending on the kind of the test and on the required precision, one of the following three solutions can be applied:

1. No correction is necessary if the rate of the barometric pressure change is equal to or slower than 1 torr/hr, the heat flux through the test sample is greater than 0.1 Btu/hr (for most evacuated powders of 1 inch or less thickness, the heat flux between room temperature and liquid nitrogen temperature will be below that figure), and a precision of $\pm$ 10% is required.
2. If the conditions described under (1) are not met, a correction in calculation can be applied using the curve of barometric pressure vs. time, mass of the cryogen in the measuring vessel at the time of the reading and properties of the cryogen at corresponding pressure.
3. The pressure stabilizer designed by ADL for Model-12 Calorimeter and described in Reference 5 can be adopted.

1.4.6 Measurement and Control of Warm Plate's Vertical Position

As we described in Section 1.3.2, the vertical position of the warm plate can be adjusted, which makes it possible

1. to use a test sample of any thickness between 0 and 1 inch;
2. to apply up to 20 psi compression to the test sample without interruption of the test.

The adjustment in height or compression is performed by manipulating the hydraulic jack located external to the sample chamber under the warm plate neck (see Figure 15). Two preloaded tension springs attached to the neck of the warm plate return the jack and the warm plate to their lowest position.

Hydraulic jack (Black Hawk, Model R618) is operated by oil. In order to keep a constant pressure on the sample for a prolonged period of time,

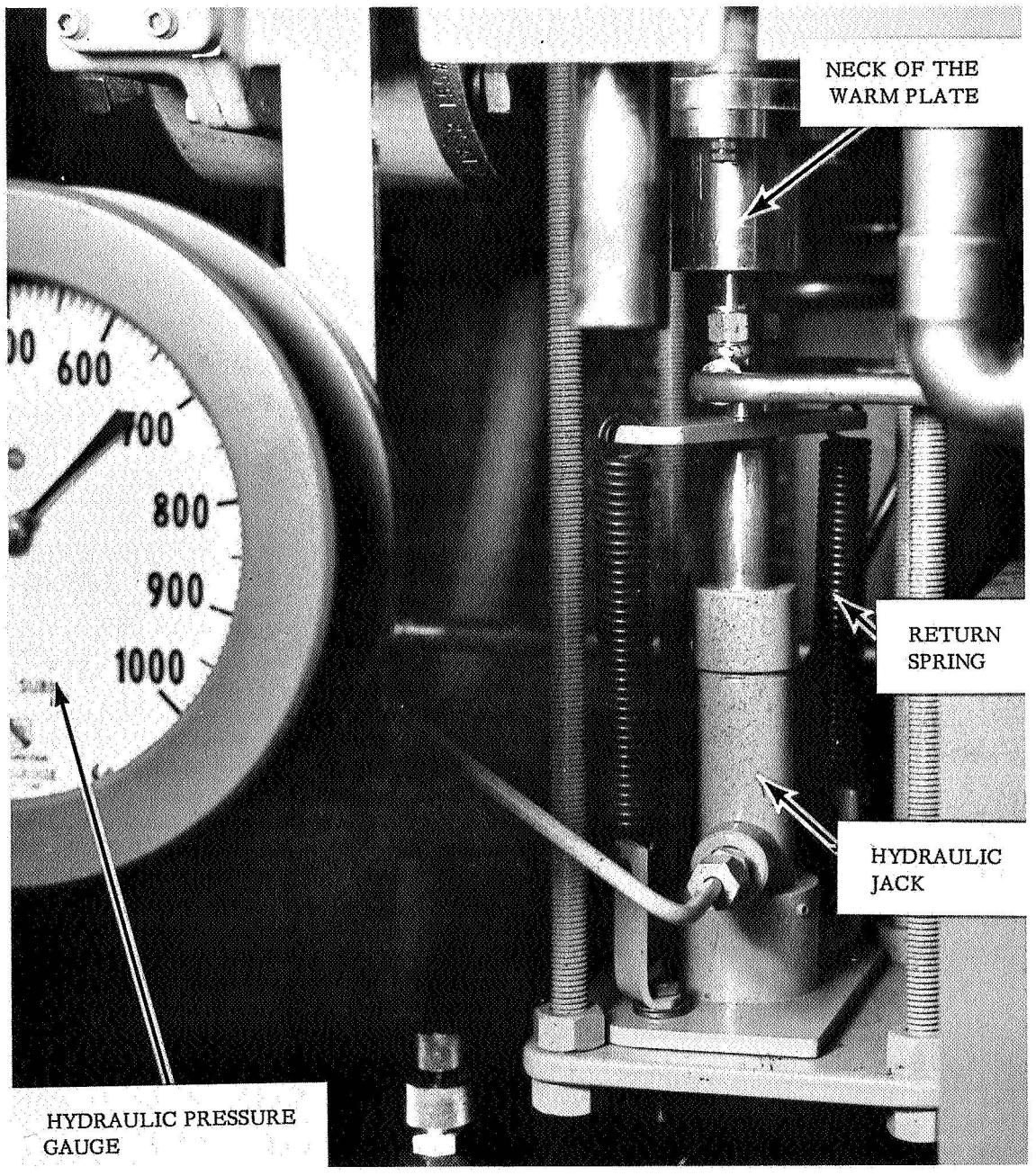


FIGURE 15 ADJUSTMENT MECHANISM FOR THE WARM PLATE POSITION

we found it necessary to have a supply of constant pressure (such as a compressed gas bottle with a regulator) acting over the oil. This arrangement provides a steady pressure on the test sample even if the test sample has a tendency to creep under compression. It is necessary to periodically calibrate the hydraulic system in order to establish the relationship between the pressure indicated by the "ram pressure" gauge (see Figure 16) and the actual compression applied to the sample. The periodic calibration is required because forces due to friction in the Teflon bearing and O-ring on the warm plate neck and in the O-ring on the hydraulic jack may vary with time, tightness of the seals, and applied lubrication.

The hydraulic jack system does not easily lend itself to a positive positioning of the warm plate. We recommend the use of three spacers equally spaced on the circumference of the warm plate. Such spacers made from Phenolic in sizes 1/4-, 3/8-, and 1/2-inch (three of each) are supplied with the calorimeter. When a pressure is applied to the hydraulic jack slightly in excess of that necessary to move the warm plate, the latter will maintain a steady position throughout the test.

1.4.7 Bell Jar and Cold Plate Lifting Hoist

The bell jar and the cold plate assembly have to be lifted in order to gain access to the sample chamber for inserting or changing of the test sample. Since the lifting is difficult to perform manually, a hydraulic hoist arrangement (see Figure 19) is provided. The hoist is operated by a pressure of city water (40-60 psig) on the lifting stroke and by its own weight on the down stroke. The three-way valve controlling the operation of the hoist is located on the right-hand side panel of the calorimeter (see Figure 13).

The same hoist can be used during a complete disassembly of the calorimeter. The bell jar can be lifted over the cold plate assembly, swung to a side, and lowered on the calorimeter table beside the base plate. Cold plate assembly then can be lifted manually.

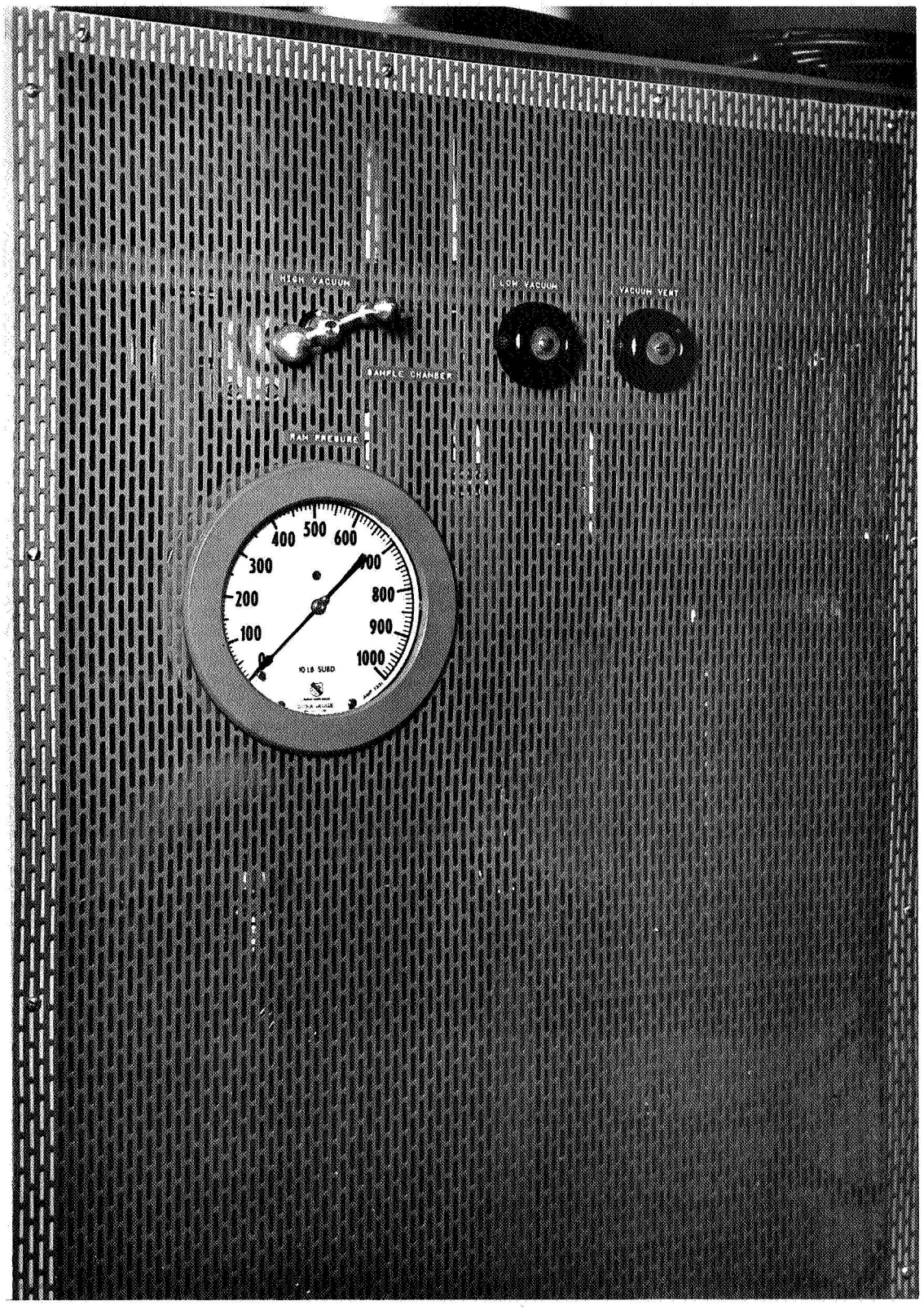


FIGURE 16 FRONT CONTROL PANEL

1.5 INSTALLATION INSTRUCTIONS

1.5.1 Position of the Calorimeter

The calorimeter should be placed in the laboratory in such a way that the right-hand control panel (Figure 13) and front control panel (Figure 16) of the apparatus are easily reachable. Enough room should be left on the left side of the calorimeter for the 4-inch high-vacuum pumping station (Section 1.4.1). Place the high-vacuum pumping station matching 4-inch flanges on the calorimeter and on the pumping station in the plan view. Then, using four jack screws provided on the base of the pumping station, lift it to match flanges in the vertical plan. Lubricate 0-ring supplied with the pumping station with a vacuum grease. To place the 0-ring into the groove on the 4-inch flange of the pumping station, swing the front of the calorimeter slightly away from the vacuum system. When swinging the calorimeter back in place, make sure the 0-ring does not fall out of the groove. Bolt the flanges together with eight 3/4-10 NCT bolts and nuts supplied with the calorimeter.

1.5.2 Service of the Constant Temperature Oil Bath

To fill the oil bath with 30 gallons of oil supplied with the apparatus, unscrew the 3/4-inch pipe plug on top of the oil bath (Figure 19). Fill oil slowly, using a funnel, or siphon oil from the cans.

1.5.3 Water and Drain Requirements

As mentioned in Section 1.4.7, the lifting hoist of the calorimeter operates on pressure of the city water. Figure 17 shows the plate where city water (in) and drain (out) are to be connected.

The high-vacuum pumping station (Section 1.4.1) requires 12 GPH of city water to cool the diffusion pump. Water and drain connections are located at low front of the vacuum station (Figure 19).

1.5.4 Electrical Requirements

Roughing pump, oil circulating pump and electrical heaters are operated by electricity. Each component is supplied with on-off switch located on the right side panel of the calorimeter (see Figure 13). The

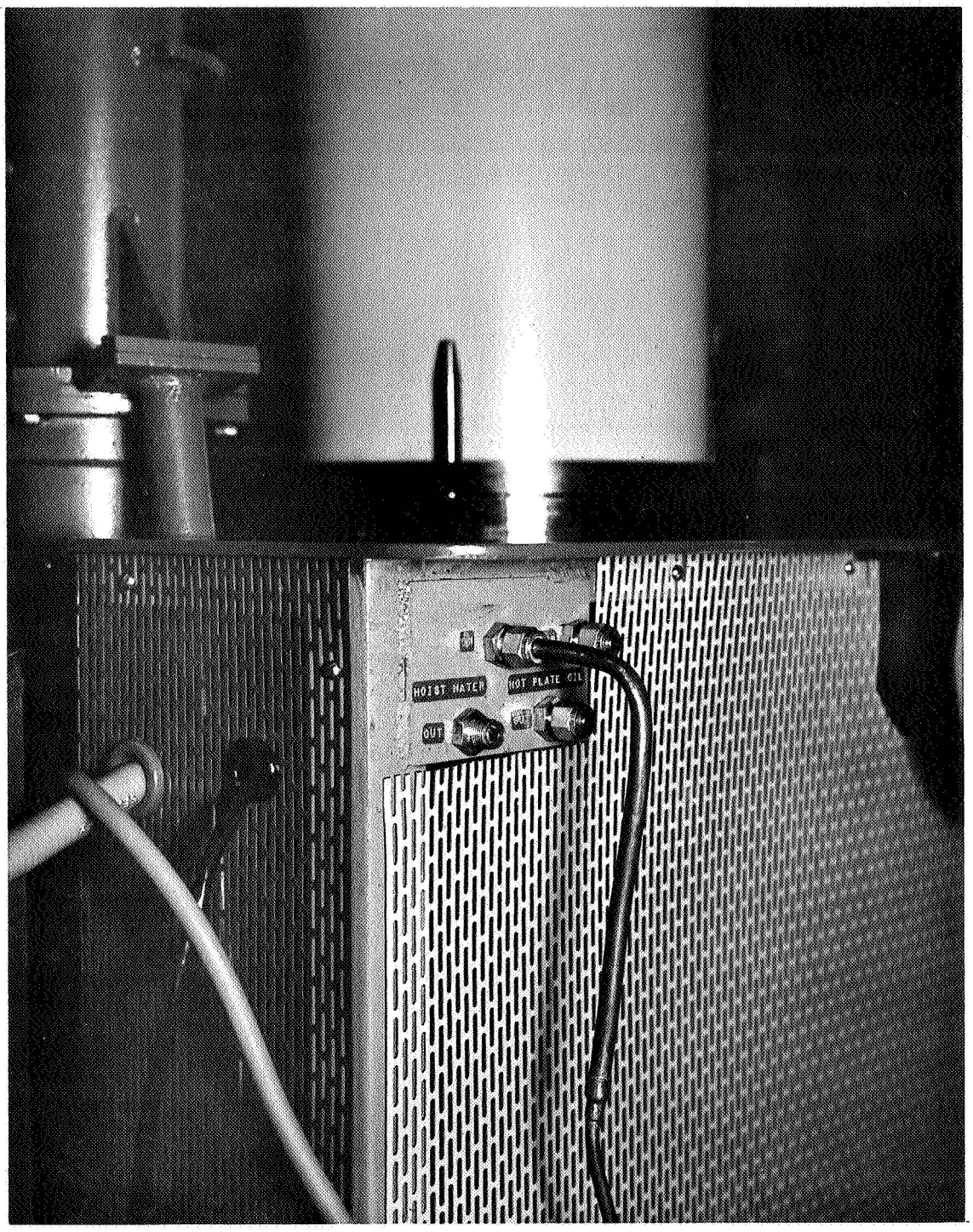


FIGURE 17 CITY WATER AND DRAINAGE CONNECTIONS FOR THE HOIST

wiring diagram of the calorimeter is presented in Figure 18. The calorimeter is wired internally, and only two 20-foot cables with standard 3-prong plugs are to be connected to a 220 V, 1-phase, 60-cycle outlet and a 110 V, 1-phase, 60-cycle outlet in the laboratory. The high-vacuum pumping station is wired internally also, and a 20-foot cable with a standard 3-prong plug is to be connected to a 110 V, 1-phase, 60-cycle outlet.

1.5.5 Requirement for Compressed Gases

As mentioned in Section 1.4.6, a constant pressure source is required to operate the hydraulic jack which positions the warm plate. A nitrogen gas bottle with 0 to 1000 psi regulator and connecting tubing and fittings is to be supplied by the customer. The supply line is to be attached to the connection on the left side of the calorimeter just below the level of the table. In addition, a nitrogen gas supply with a low-pressure regulator (below 1 psig) is necessary for breaking vacuum in the bell jar and sample chamber after each test. We also advise having a low-pressure helium supply handy for occasional vacuum leak detection work.

1.6 PROCEDURE FOR CHANGE OF TEST SAMPLE

1.6.1 Disassembly of the Calorimeter (Follow Figure 19)

Before disassembly is started, all of the steps in Sections 1.7.5 and 1.7.6 must have been carried out.

1. Remove all 6 bolts holding the bell jar to the base plate.
2. Remove all four 1/4-inch O.D. gum rubber tubes leading from the top of measuring-guard vessel to the corresponding instrumentation.
3. Swing rubber tube support out of the way of the rising bell jar.
4. Remove 1/4-inch O.D. gum rubber sleeves over the four fill and vent lines to the measuring and guard vessels.
5. Remove three metal clamps tightening the 2-inch I.D. rubber sleeve placed over the bell jar neck.
6. Pull out the guard vessel neck enclosure.

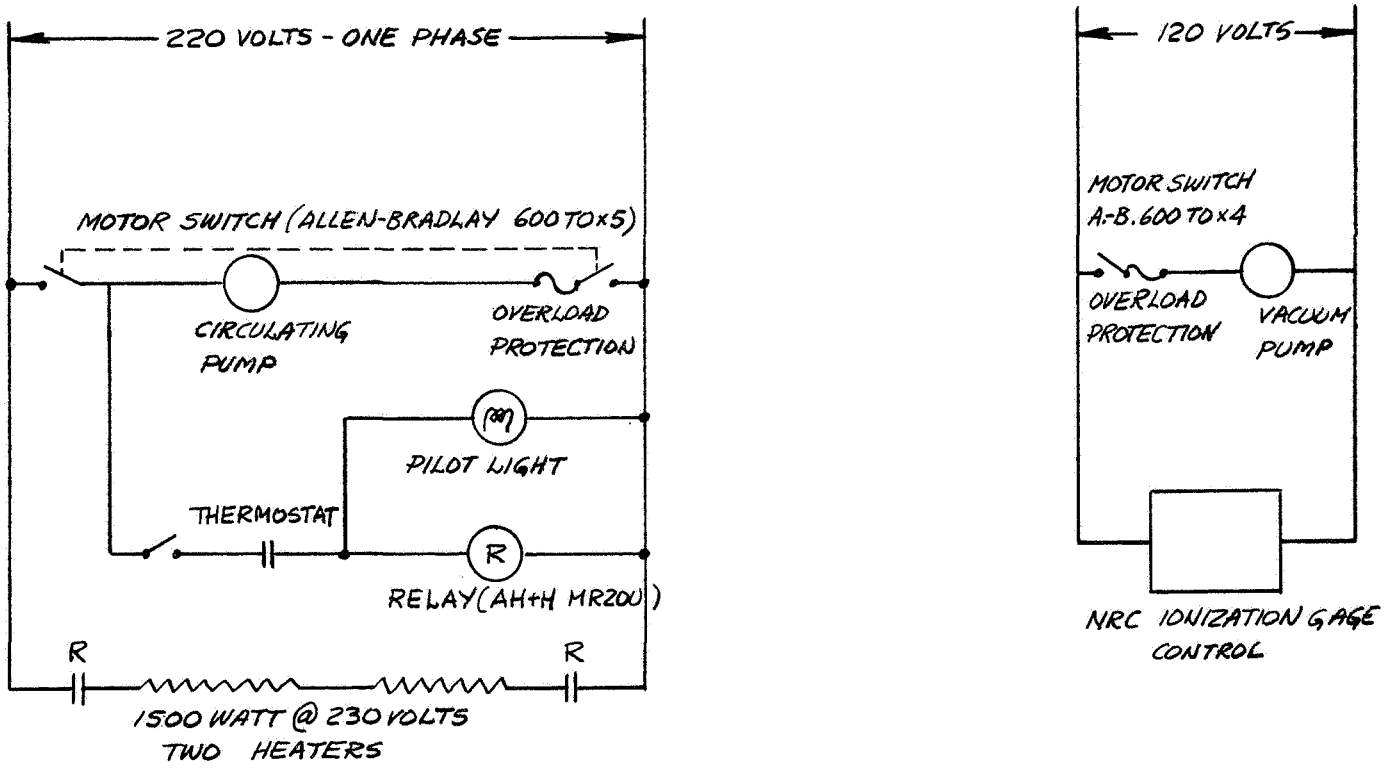


FIGURE 18 WIRING DIAGRAM

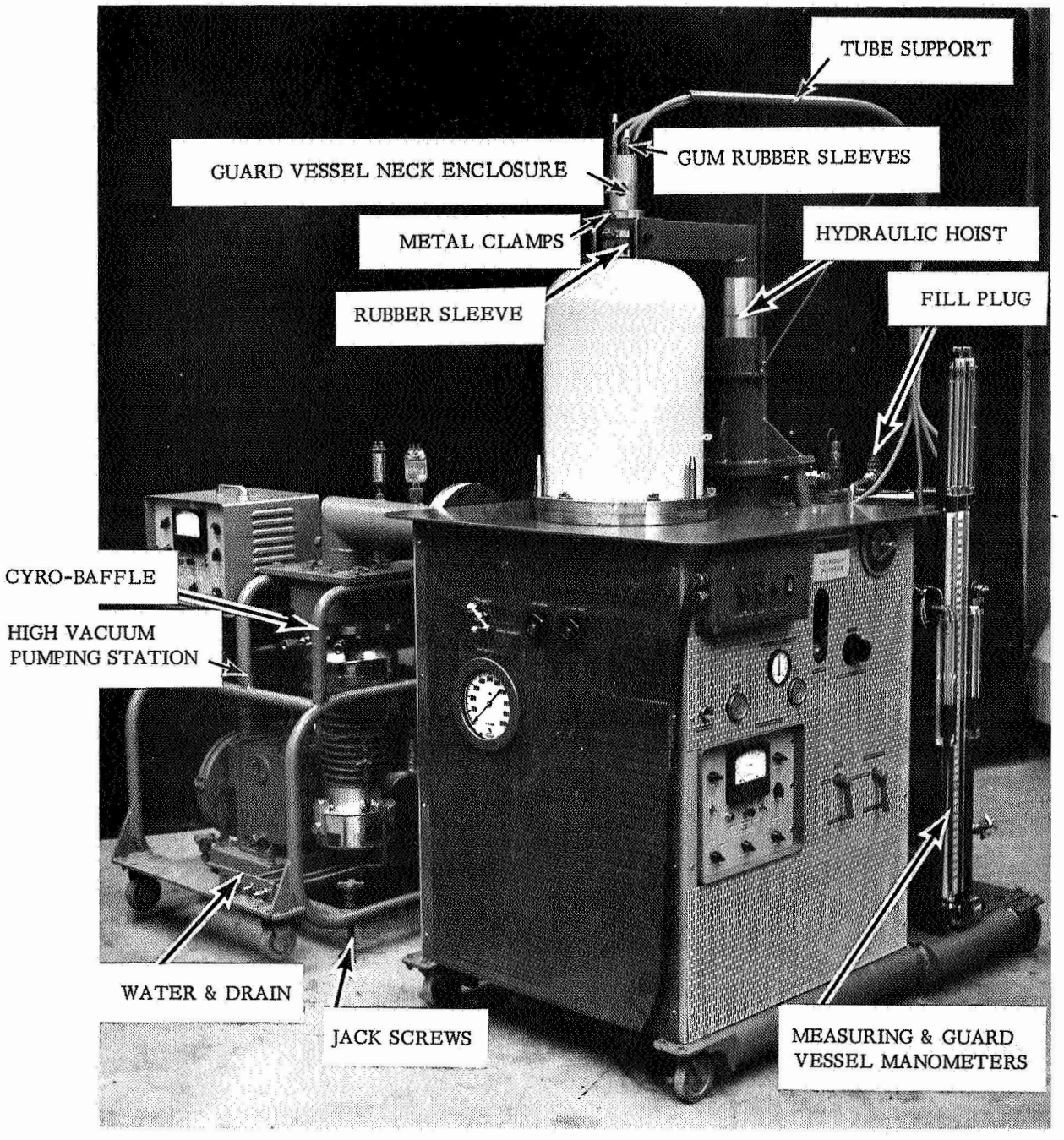


FIGURE 19 GUIDE TO ASSEMBLY AND DISASSEMBLY OF THE CALORIMETER

7. Remove the 2-inch I.D. rubber sleeve.
8. Lift slowly the bell jar to the position just above the flange of the cold plate assembly (see Figure 20) by operating the hydraulic hoist valve (see Figure 13). Attention: Position of the hoist valve handle determines the rate of lifting.
9. Remove all 12 bolts holding the cold plate assembly to the sample chamber flange.
10. Insert 3 cold plate lifting ears into holes in the cold plate flange (see Figure 20).
11. Lower the bell jar slowly into the position just above (1/16" to 1/32") the lifting ears.
12. Use any three bolts supplied to hold the bell jar to the base plate for securing the lifting ears to the bell jar flange (see Figure 21).
13. Lift the bell jar-cold plate assembly.
    Attention: Only for complete disassembly of the calorimeter or for a study of the test sample is it necessary to lift the assembly above the ends of the cold plate guide pins (as shown in Figure 21). Then the assembly can be swung out of the way on the hoist arm and lowered to rest on the calorimeter table. During the normal sample change, it is not recommended to lift the assembly above the ends of the guide pins.
14. Remove ths S.S. diaphragm (if used) and the O-ring (if used) on top of the sample chamber.
15. Examine and remove the test sample.
16. Check sample chamber for cleanliness.
17. The calorimeter is ready for a new test sample.

1.6.2 Reassembly of the Calorimeter

Before reassembly is started, all of the steps shown in Section 1.6.1 must have been carried out. Follow Figure 21, then 20, then 19.

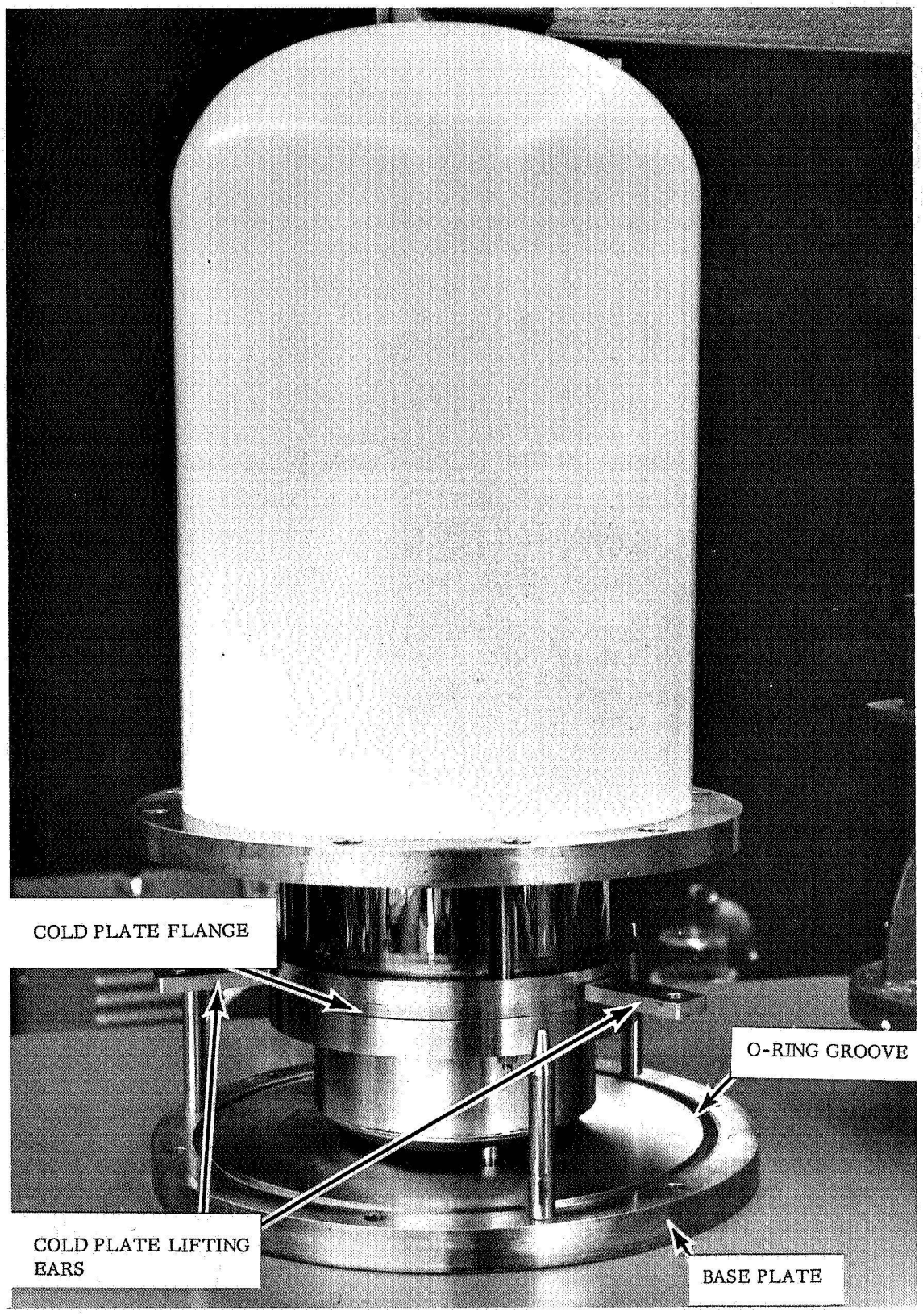


FIGURE 20 LIFTING OF THE BELL JAR

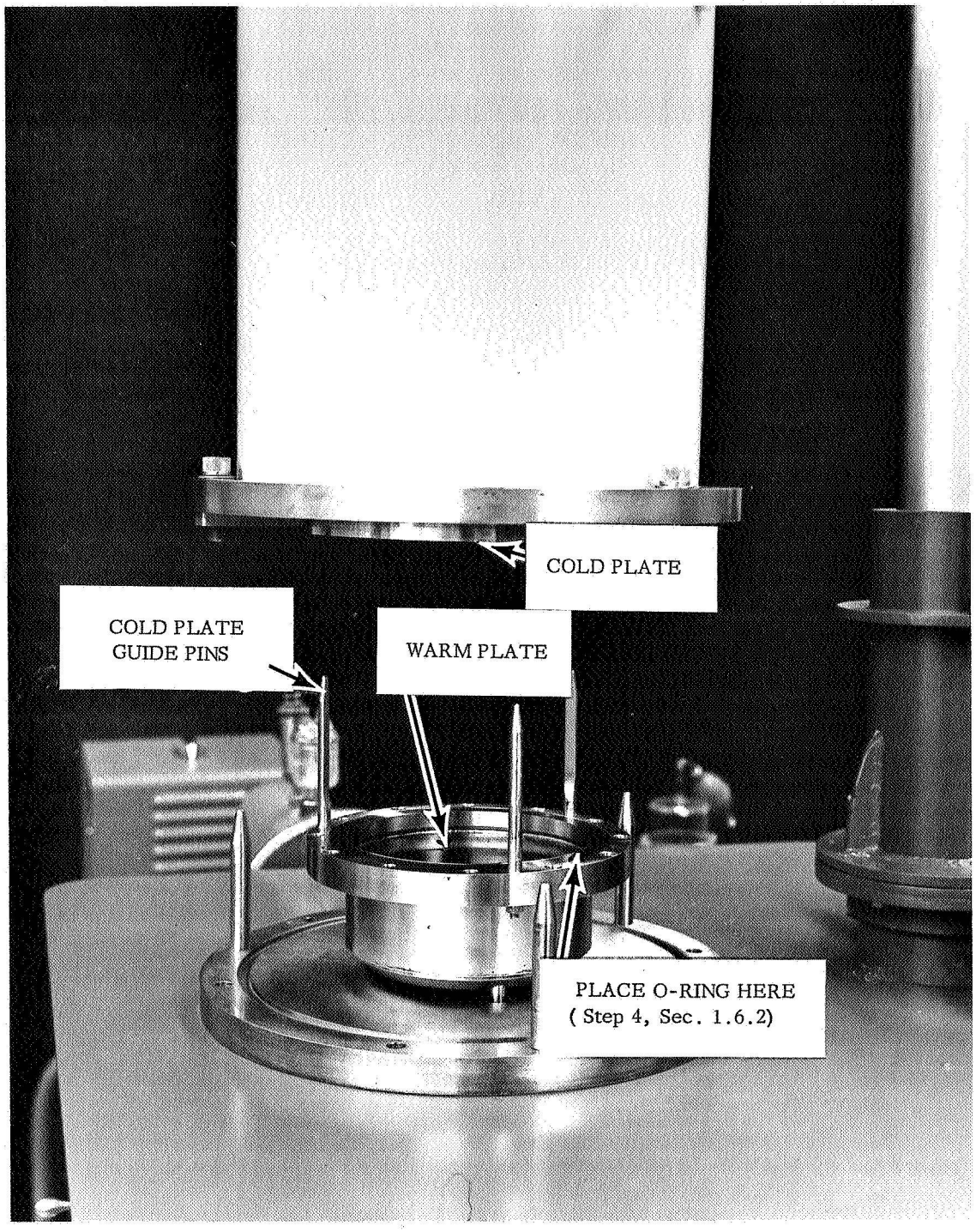


FIGURE 21 LIFTING OF THE COLD PLATE ASSEMBLY

1. Examine the surfaces of the cold and warm plate to make sure that the black paint is in good condition. Repaint if necessary (see item 6, Section 1.8.4 for paint specifications).
2. Set the polyester film ring at the edge of the warm plate into a required position if a powder insulation is to be tested. Remove it, if other than powder will be tested.
3. Place the test sample on the warm plate.
4. Place an 0-ring on the sample chamber flange (item 4, Section 1.8.4) and a new S.S. diaphragm (item 5, Section 1.8.4) over the 0-ring (only if required).
   Attention: The use of S.S. diaphragm may supply another possible source of error in the measurement of the true heat flux through the test sample; therefore, the use of the 0-ring is recommended only in cases where the sample chamber pressure has to be other than that of the bell jar at any time during the test.
5. Lower slowly the cold plate, watching that the 0-ring and the S.S. diaphragm stay in place.
6. Bolt the cold plate to the sample chamber flange with all 12 bolts. Tighten all bolts evenly.
7. Remove three bolts holding the lifting ears to the bell jar flange.
8. Remove and store the three lifting ears (Figure 20).
9. Make sure the 0-ring in the base plate is properly greased and located in the groove.
10. Lower slowly the bell jar.
    Attention: Make sure that
    a. the bell jar does not hang on the edge of the guard vessel neck;
    b. the bell jar does not hang on the tips of the bell jar guiding pins;
    c. the 0-ring in the groove of the base plate is still properly located just before the bell jar touches it.

11. Bolt the bell jar to the base plate. Tighten all bolts evenly (Figure 19).
12. Grease and replace the 2-inch I.D. rubber sleeve over the guard vessel and bell jar necks.
13. Replace the guard vessel neck enclosure, fitting it under the rubber sleeve placed in step 12. The end of the enclosure should be approximately 3/4 inch down inside the sleeve.
14. Replace all three metal clamps over the rubber sleeve. Make sure that the clamps are tight so that no gas can enter the bell jar or the guard vessel.
15. Replace four gum rubber sleeves over the 1/4-inch O.D. vent and fill lines closing the gaps between the lines and the guard vessel neck enclosure.
16. Swing the rubber tube support into its proper position. Attention: Do not replace the four 1/4-inch O.D. gum rubber tubings over the respective vent lines from the measuring and guard vessels.
17. The unit is assembled and ready for evacuation.

1.7 TEST PROCEDURE

1.7.1 Evacuation of the Bell Jar and Sample Chamber

Before this procedure is started, all of the steps in Sections 1.6.2 and 1.7.6 must have been carried out.

At the outset of this sequence, the vacuum system may be off completely, in which case the procedure as written below may be followed verbatum; or the high vacuum pumping station and/or roughing pump may be operating, in which case the steps that specify turning on a component that is already on may be ignored.

1.7.1.1 High Vacuum in the Bell Jar and the Sample Chamber

1. Turn on the water that cools the diffusion pump on the high vacuum station (see Sections 1.4.1 and 1.5.3).
2. Turn on forepump on the high vacuum pumping station.

3. Turn on roughing pump on the calorimeter (switch marked "vacuum pump" in Figure 13).
4. Turn on the power switch at both thermocouple-ionization gauge controls and switch them to read thermocouple gauges. (See Figure 13, right hand toggle switch in up position, left hand lower selector switch on "TC-1".)
5. Open the 2-inch gate valve marked "high vacuum" in Figure 16.
6. Open slowly the 1-inch block valve marked "low vacuum" in Figure 16.

   Attention: Make sure the block valve marked "vacuum vent" (Figure 16) is tightly closed.
7. When the pressure indicated on the forepump thermocouple gauge of the high vacuum pumping station (Figure 19) is below 25 microns, switch on power to the diffusion pump.
8. Wait one-half hour until diffusion pump is warmed up.
9. If the pressure indicated on the sample chamber thermocouple gauge is below 50 microns, close the 1-inch block valve marked "low vacuum" in Figure 16.
10. Open the 4-inch gate valve on the high-vacuum pumping station (see Figure 22). The pressure should drop below 5 microns within 1/2 hour.
11. Fill cryo-baffle on the high vacuum system with liquid nitrogen (Figure 19).

    Attention: Refill cryo-baffle as required during the test. Experience showed that the liquid stays in the baffle for approximately 2 hours.
12. Turn on ionization gauges for the sample chamber and the bell jar (for steps in the procedure see the NRC instruction book supplied with the calorimeter) when the corresponding thermocouples show well below 5 microns.
13. When the pressure in the bell jar is below 5×10^{-5} torr, evacuation of the bell jar and sample chamber is completed. It may take 1 to 8 hours to reach this pressure, depending upon the test sample used and on the cleanliness of the vacuum system.

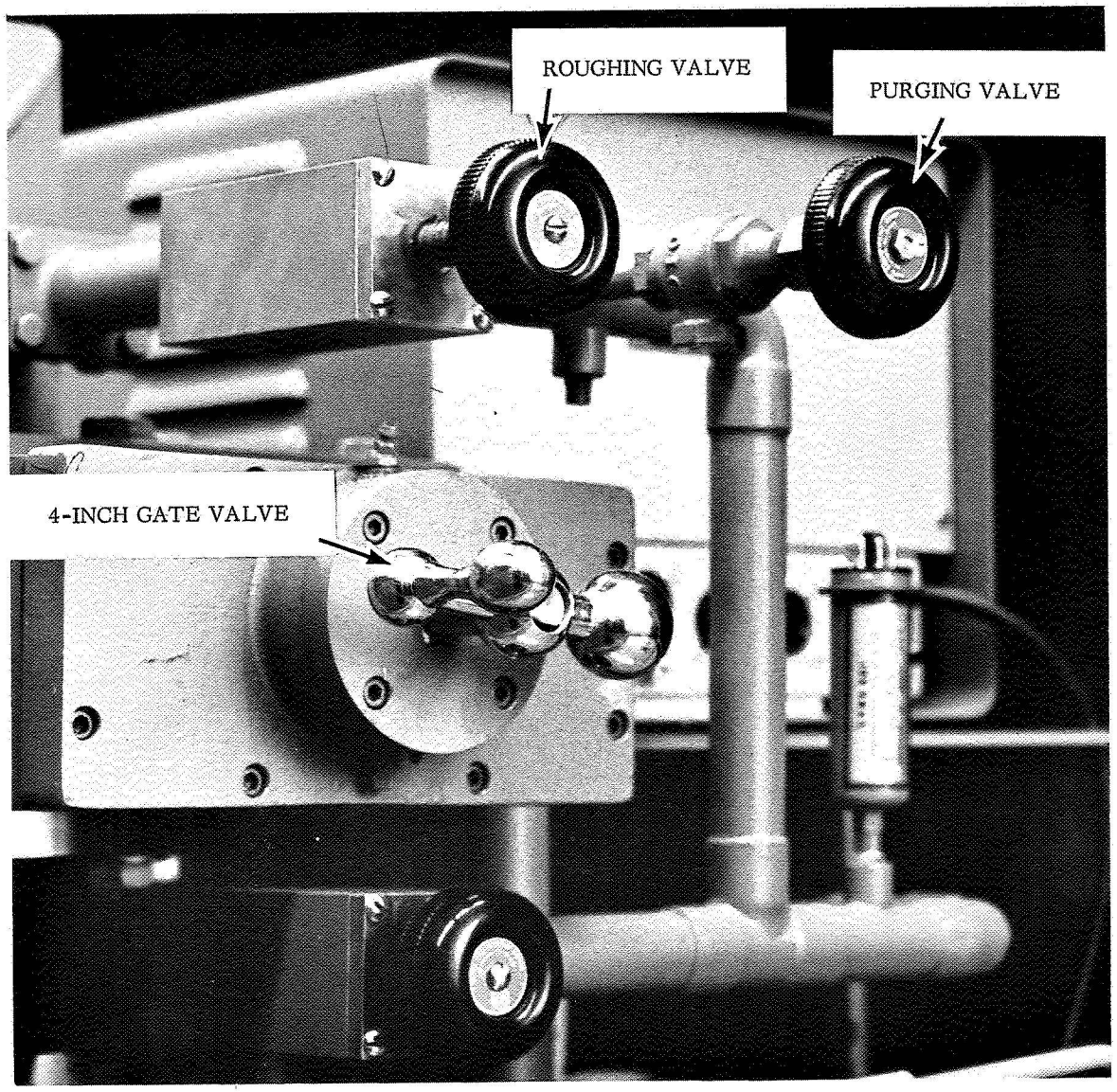


FIGURE 22 VACUUM CONTROL VALVES FOR THE HIGH VACUUM PUMPING STATION

14. The calorimeter is ready for liquid nitrogen transfer into the guard and measuring vessels.

1.7.1.2 High Vacuum in the Bell Jar and Pressure Other Than High Vacuum in the Sample Chamber

In order to maintain a pressure in the sample chamber that is independent of the pressure in the bell jar, a sealed diaphragm must be used to isolate the chambers. (See Section 1.3.3 and step 4, Section 1.6.2.)

Perform steps 1 through 13 as in Section 1.7.1.1. The sample chamber is then evacuated. Close the 2-inch gate valve marked "high vacuum-sample chamber" in Figure 16. The desired gas at predetermined pressure may be introduced into the sample chamber through the 1-inch block valve marked "vacuum vent" in Figure 16.

1.7.2 Adjusting of the Warm Plate Temperature (Follow in Figure 13)

1. Start oil circulating pump by flipping toggle switch marked "oil pump".
2. Place selector valve marked "hot plate fluid" on "sample chamber".
3. Place "bypass valve" on "closed".
4. Observe oil flow on the "oil flow" indicator.
5. If a warm plate temperature higher than room temperature is desired, set the thermostat placed over the oil bath to the required temperature.
6. Start oil heater by flipping switch marked "oil heater". (The light next to the switch should go on. When the temperature set on the thermostat is reached, the light will blink.)
7. Observe the temperature in the warm plate on dial thermometers marked "in" and "out".

1.7.3 Filling the Measuring and Guard Vessels with Cryogen

Before this procedure is started, all of the steps in Section 1.7.1.1 or 1.7.1.2 and 1.7.2 must have been carried out.

1. Connect one vent of the measuring vessel with one vent of the guard vessel with short pieces of 1/4-inch I.D. gum rubber tubing. Do the same with two other vents.
2. Connect cryogen storage dewar with the fill line of the guard vessel.
3. Open the valve on the storage dewar and start transfer of the cryogen.
4. Stop transfer when liquid begins to shoot from the measuring vessel fill.
5. Disconnect both pieces of gum rubber tubing placed in step 1.
6. Disconnect the liquid transfer line from the fill line of the guard vessel and cap the fill line.
7. Connect the liquid transfer line to the fill of the measuring vessel.
8. Open the valve on the storage dewar and start transfer of the cryogen.
9. Stop transfer when liquid begins to shoot from both measuring vessel vents.
10. Disconnect the transfer line from the fill line of the measuring vessel and cap the fill line.
11. Let both vessels boil for 1/2 hour.
12. Repeat 2 and 3.
13. Stop transfer of the liquid when the liquid begins to shoot from both vents of the guard vessel.
14. Wait a few minutes, then open the transfer valve slowly, allowing liquid to barely trickle from the vents for a few seconds.

    Attention: Prolonged exposure of the guard vessel neck to liquid nitrogen during transfer may cause freezing of the 2-inch I.D. rubber sleeve on the guard vessel neck, with subsequent loss of vacuum. A hot air gun turned on the rubber sleeve normally prevents the freezing.

1.7.4 Taking Data

When the transfer of cryogenic liquid into the measuring vessel and guard vessel is complete, the next task is to determine when thermal equilibrium has been established, both in the sample and in the cryogen, and to measure the heat flux into the measuring vessel at that state. This is best accomplished by measuring the boil-off rate continuously over a period of time. The boil-off rate typically will be high after initial filling with cryogen, will decrease when excess heat from the sample and apparatus has been carried off, and will reach a steady rate when thermal equilibrium has been established. Insulation systems that have a low thermal conductivity, such as evacuated powders, require several hours to reach equilibrium, while relatively poor insulators, such as foams in air, may stabilize in less than two hours. During this time it is necessary to maintain a constant pressure on the liquid in the measuring vessel to prevent errors due to enthalpy changes. It is also necessary to keep the guard vessel pressure constant and nearly the same as that in the measuring vessel in order to minimize temperature differences and hence heat transfer between them. The guard vessel pressure should, however, be slightly higher than the measuring vessel pressure, in order to avoid recondensation of the boil-off gas as it passes by the liquid in the guard vessel. We find it convenient to maintain the guard vessel pressure within plus 5 mm minus 0 mm of the measuring vessel pressure. This task can be accomplished by positioning the vent line discharge nozzle from the guard vessel higher or lower in the oil-filled graduate supplied for this purpose.

If reaching equilibrium takes more than six hours, the investigator is well advised to refill the guard vessel, then, again, wait for equilibrium which normally would be reached in one to two more hours.

The length of the data taking period is entirely up to the investigator. As a guide line, we suggest a minimum of 2 hours with minimum of 4 data points taken during this time. For samples exhibiting low heat fluxes, such as 1/2 to 1-inch thick evacuated powders, 6 to 8 hour long tests (after equilibrium is reached) are recommended.

A suggested form for taking data is given in Section 1.8.1. Table I in the same section gives an explanation of the nomenclature used on that

data form. Section 1.8.2 presents a sample calculation. The intervals at which this data should be recorded depend upon the boil-off rates observed. In case the phenolic spacers (see Section 1.4.6) are not used, the distance between the warm and the cold plates, which in most cases will be identical to the sample thickness, may be measured by gauging the distance that the stub of the warm plate neck protrudes from the sample chamber neck (Figure 15). We recommend that this measurement be made at the beginning and at the end of each test.

1.7.5 Removal of the Cryogen from the Calorimeter and Warm-Up Procedure

This procedure should be followed at the completion of each test.

1. Switch off both ionization gauges.
2. Turn both ionization gauge controls to read thermocouples.
3. Disconnect all four 1/4 I.D. gum rubber tubings from the vent lines of the measuring and guard vessels (Figure 19).
4. Remove stoppers from the fill lines of the measuring and guard vessels and slip them over one of the vents for each vessel.
5. Slip short pieces of gum rubber tubing over the fill lines of the measuring and guard vessels to divert the flow of the cryogen into a chosen direction.
6. Pressurize both measuring and guard vessels with nitrogen gas using the vents which were left open in step 2. (Do not exceed 10 psig.)
7. Keep the pressure on both vessels until all cryogenic liquid is blown from both vessels.
8. Reduce pressure to a few inches of water above atmospheric and cap all inlets to the vessels with rubber policemen. Attention: Rubber policemen will not permit air and water condensation inside the vessels, simultaneously providing a safety valve should the pressure inside the vessel increase during warm-up cycle.
9. Close all vacuum valves leading to the bell jar (4-inch gate valve on the high vacuum system, see Figure 22) and to the

sample chamber (marked "high vacuum" and "low vacuum", in Figure 16).

10. Release the pressure on the hydraulic jack, permitting the warm plate to move down (observe pressure gauge shown in Figure 16).
11. Adjust the pressure regulator on the nitrogen gas bottle to below 1 psig. Purge the supply line with dry nitrogen from the bottle.
12. Connect the bottle to the valve marked "vacuum vent" (Figure 16).
13. Crack slowly the "vacuum vent" valve to break vacuum in the bell jar and the sample chamber.
14. Observe first the thermocouple gauge indication; then the indication of the compound gauge (marked "sample chamber pressure" in Figure 13) until sample chamber pressure becomes atmospheric (0 on the compound gauge).
15. Turn off both thermocouple-ionization gauge controls.
16. Turn off oil heater but leave the circulation pump going in order to prevent freezing of the oil in the warm plate.
17. Turn off the oil circulating pump approximately 2 hours after the cryogenic liquid was blown out.
18. Let the calorimeter warm up for at least 6 hours (preferably overnight) before proceeding to disassemble it (see Section 1.6.1).

Note: If the next test is planned to be run right after change of the sample, we recommend that both the high vacuum pumping station and the vacuum pump on the frame of the calorimeter be allowed to run.

1.7.6 Shutdown of the Vacuum System

The system may be completely shut down or the high vacuum pumping station may be isolated and allowed to operate while the remainder of the system is shut down.

1.7.6.1 Complete Shutdown

Make sure that steps 9 and 15 of Section 1.7.5 are performed.

1. Turn off the switch marked "vacuum pump" in Figure 13; this cuts off the roughing pump mounted on the calorimeter frame. Vent the pump.
2. Turn off diffusion pump on the high vacuum pumping station.
3. Wait 1/2 hour and turn off forepump of the high vacuum pumping station.
4. Bleed air or nitrogen into the system by opening purge valve (Figure 22).
5. Valve off the water cooling to the diffusion pump.

1.7.7 Storing of the Calorimeter

If the calorimeter is to be inoperative for a short period of time, the components should be arranged as follows:

1. The bell jar is to be closed to act as a dust cover for the cold plate and the vacuum space.
2. All four vent and fill connections to both vessels in the cold plate are to be capped with hose policemen.
3. All valves to the bell jar and sample chamber are to be closed.
4. All electrical equipment is to be turned off.
5. The bell jar and sample chamber are to be filled with nitrogen gas.
6. The pressure on the warm plate hydraulic jack is to be released.

1.8.1 Data Sheet for Use with ADL Model-6 Calorimeter

Test No. 1
Test Date: January 20, 1965
Investigator: R. Carroll
Test Variable: Acceptance Test
Sample Description: Pumice Powder, 62-125 microns
Sample Weight: 143 gr.

Time of Day	Elapsed Time	Displaced Volume	Boil-Off Gas Temperature	Barometric Pressure	Cold Plate Temperature	Warm Plate Temperature			Pressure		Compression on the Sample	Pressure		Distance Between Plates
						Dial Thermometers		Thermo-Couples						
						In	Out		Bell Jar	Sample Chamber		Measuring Vessel	Guard Vessel	
a.m. p.m.	hrs.	cm^3 or ft^3	^oF	torr	^oF	^oF	^oF	^oF	torr	torr	psi	mm of H_2O	mm of H_2O	inch
(1)	(2)	(3) cm^3	(4)	(5)	(6)	(7)	(8)	(9)	(10)	(11)	(12)	(13)	(14)	(15)
	0	200	79	751.5	-320	-	-	112						.297
	58/60	2000	28	752.0	-320	-	-	112						.297

TABLE I EXPLANATION OF NOMENCLATURE

Column	Symbol	Units	Explanation
1	--	hrs:min	Time of day for reference.
2	t	hrs	Total elapsed time from beginning of the run.
3	V	cm^3 or ft^3	Total displaced volume indicated by wet test meter in cubic feet or by low boil-off meter in cubic centimeters.
4	T_G	^oF	Gas temperature as indicated by a thermometer on the wet test meter or room temperature if low boil-off meter is used.
5	P_B	torr	Barometric pressure at the time of the reading.
6	T_c	^oF	Cold plate temperature, same as the boiling temperature of the cryogen in the cold plate.
7	--	^oF	Temperature of oil on the inlet side of the warm plate, as indicated by a dial thermometer (Figure 13).
8	--	^oF	Temperature of oil on the outlet side of the warm plate, as indicated by a dial thermometer (Figure 13).
9	T_w	^oF	Warm plate temperature, the average reading of four thermocouples embedded in the warm plate.
10	P_{BJ}	torr	Vacuum in the bell jar as indicated by the ionization gauge control.
11	P_{SC}	torr	Vacuum or pressure in the sample chamber as indicated by the thermocouple-ionization gauge control or by the compound dial gauge.
12	F	psi	Mechanical compression on the test sample applied by the hydraulic jack.
13	--	mm H_2O	Pressure over the cryogen in the measuring vessel as indicated by the oil manometer.

TABLE I (cont'd)

Column	Symbol	Units	Explanation
14	--	mm H_2O	Same for the guard vessel.
15	L	inch	Distance between cold and warm plates (in most cases same as sample thickness). Indicated by the thickness of the phenolic spacers used or by the length of the warm plate neck protruding below the O-ring gland.

1.8.2 Sample Calculations

1. Remarks:

   Omit steps 2 and 3 if a wet test meter indicating in cubic feet is used.

2. For low boil-off meter only:

   Conversion of the boil-off volume into cubic feet

$$V\left[ft^3\right] = 3.53 \times 10^{-5}\ V$$

   where: $V\left[cm^3\right]$ - boil-off volume (entry in Column 3)

   $V_1 = 200\ cm^3$; $V_1 = 3.53 \times 10^{-5} \times 200 = 7.06 \times 10^{-3}\ ft^3$

   $V_2 = 2000\ cm^3$; $V_2 = 3.53 \times 10^{-5} \times 2000 = 70.6 \times 10^{-3}\ ft^3$

3. For low boil-off meter only:

   Height of the oil in the graduate at the time of the reading. Refer to Figure 23.

   $V_1 = 200\ cm^3$ $\qquad$ $H_1 = 16.74$ inches

   $V_2 = 2000\ cm^3$ $\qquad$ $H_2 = 1.80$ inches

4. Pressure correction

   a. For pressure drop in the wet test meter

$$\Delta P\left[torr\right] = \frac{25.4}{13.6}\ h = 1.86h$$

   where: 25.4 - conversion from inches to millimeters

   13.6 - specific gravity of mercury

   h [inches] - pressure drop indicated by water manometer on the wet test meter

   b. For the height of the oil in the graduate of the low boil-off meter at the time of the reading

$$\Delta P\left[torr\right] = \frac{0.8 \times 25.4}{13.6}\ H = 1.49H$$

   where: 0.8 - specific gravity of oil

   13.6 - specific gravity of mercury

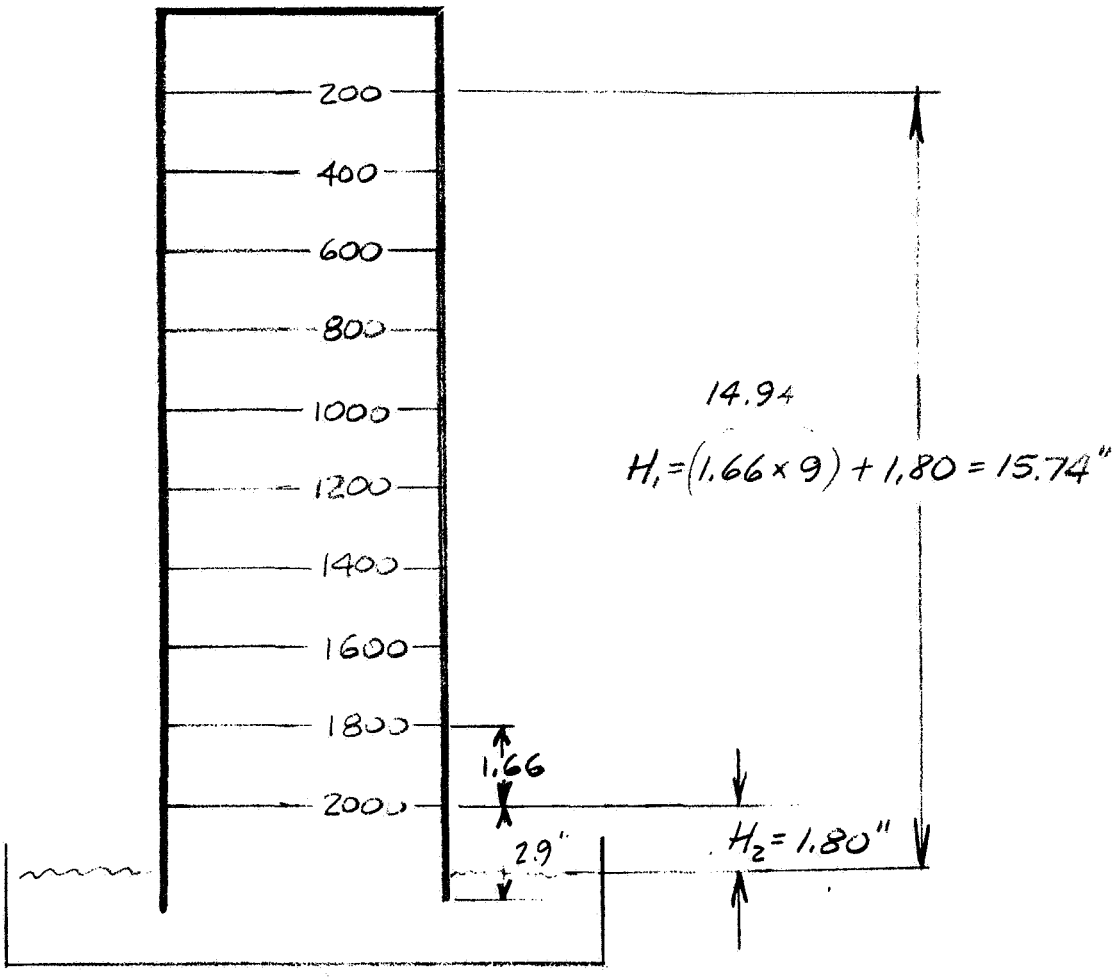


FIGURE 23 HEIGHT OF OIL IN THE GRADUATE AT V_1=200 CM^3 ; & V_2= 2000 CM^3

25.4 - conversion factor $\left(\frac{\text{mm}}{\text{inch}}\right)$

H [inch oil] - height of oil, calculated in step 3.

$H_1 = 16.74$ inches $\quad \Delta P_1 = 1.49 \times 16.74 = 25.1$ torr

$H_2 = 1.80$ inches $\quad \Delta P_2 = 1.49 \times 1.80 = 2.7$ torr

5. Reduction of the boil-off volume to the standard conditions ($0^{\circ}C$ and 760 torr)

$$V_{ST}\left[\text{ST ft}^3\right] = V \times \underbrace{\frac{492}{460 + T}}_{\text{temperature correction}} \times \underbrace{\frac{P_B - \Delta P}{760}}_{\text{pressure correction}}$$

where: V [ft^3] - boil-off volume in ft^3 calculated in step 2

T [$^{\circ}F$] - boil-off gas temperature (entry in Column 4)

P_B [torr] - barometric pressure (entry in Column 5)

ΔP [torr] - pressure correction explained in step 4

$$V_{ST_1} = 7.06 \times 10^{-3} \underbrace{\frac{492}{460 + 79}}_{539} \times \underbrace{\frac{751.5 - 25.1}{760}}_{726.4} = 6.2 \times 10^{-3} \text{ ST ft}^3$$

$$V_{ST_2} = 70.6 \times 10^{-3} \frac{492}{460 + 78} \times \underbrace{\frac{752.0 - 2.7}{760}}_{749.3} = 63.8 \times 10^{-3} \text{ ST ft}^3$$

6. Calculation of the true boil-off rate

$$\dot{V}_{ST}\left[\frac{\text{ST ft}^3}{\text{hr}}\right] = \frac{V_2 - V_1}{t_2 - t_1}$$

where: V_2, V_1 [ST ft^3] - boil-off volumes, calculated in step 5

t_2, t_1 [hrs] - elapsed time (entries in Column 2)

$V_2 = 63.8 \times 10^{-3}$ ST $\text{ft}^3 \qquad t_2 = 58/60$ hr

$V_1 = 6.2 \times 10^{-3}$ ST $\text{ft}^3 \qquad t_1 = 0$

$$\dot{V}_{ST} = \frac{63.8 - 6.2}{58/60 - 0} = \frac{57.6 \times 60}{58} \times 10^{-3} = .0596 \frac{\text{ST ft}^3}{\text{hr}}$$

7. Heat flux passing through the test sample (for cold plate at liquid nitrogen temperature)

$$\dot{Q}\left[\frac{Btu}{hr}\right] = 85.7 \times 0.0781 \times \dot{V}_{ST} = 6.7 \times \dot{V}_{ST}$$

where: $85.7 \frac{Btu}{lb}$ - heat of vaporization of one lb of liquid nitrogen

$0.0781 \frac{lb}{ST\ ft^3}$ - density of liquid nitrogen

$\dot{V}_{ST}\left[\frac{ST\ ft^3}{hr}\right]$ - true boil-off rate, calculated in step 6

$\dot{V}_{ST} = .0596 \frac{ST\ ft^3}{hr}$; $\dot{Q} = 6.7 \times .0596 = .399 \frac{Btu}{hr}$

8. Apparent thermal conductivity of the test sample

$$K\Big|_{T_c}^{T_w}\left[\frac{Btu\text{-}in}{^oF\ hr\ ft^2}\right] = \frac{\dot{Q}L}{A(T_w - T_c)}$$

where: $\dot{Q}\left[\frac{Btu}{hr}\right]$ - heat flux through the sample, calculated in step 7

$L\left[inch\right]$ - thickness of the sample (entry in Column 15)

$A\left[ft^2\right]$ - area of the measuring vessel in contact with the test sample

$A = .0642\ ft^2$ (d_{MV} = 3-7/16 inch)

$T_w\left[^oF\right]$ - average temperature of the warm plate (entry in Column 9)

$T_c\left[^oF\right]$ - temperature of the cold plate (entry in Column 6)

$\dot{Q} = .399 \frac{Btu}{hr}$; $L = .297$ inch; $A = .0642\ ft^2$

$T_w = 112^oF$; $T_c = -320^oF$

$$K\Big|_{-320}^{112} = \frac{.399 \times .297}{.0672(112 + 320)} = 4.1 \times 10^{-3} \frac{Btu\text{-}in}{^oF\ hr\ ft^2}$$

$$432$$

9. Conversion of thermal conductivity into various systems of measurement (see Table II).

$$K\Big|_{-320}^{112} = 4.1 \times 10^{-3} \frac{\text{Btu-in}}{{}^{o}\text{F hr ft}^2} = 1.44 \times 10^{-3} \times 4.1 \times 10^{-3}$$

$$= 6 \times 10^{-6} \frac{\text{watt}}{\text{cm}\,{}^{o}\text{C}}$$

TABLE II CONVERSION FACTORS FOR THERMAL CONDUCTIVITY

$\frac{\text{Btu-in}}{\text{hr ft}^2\,{}^{o}\text{F}}$	$\frac{\text{Btu}}{\text{hr ft}\,{}^{o}\text{F}}$	$\frac{\text{g-cal.}}{\text{sec cm}^2\,{}^{o}\text{C}}$	$\frac{\text{watt}}{\text{cm}\,{}^{o}\text{C}}$	$\frac{\text{kg-cal.}}{\text{hr m}\,{}^{o}\text{C}}$
1	.0833	.000345	.00144	.124
12	1	.00413	.0173	1.49
2900	242	1	4.19	360
693	57.8	.239	1	86
8.06	.672	.00278	.0116	1

1.8.3 Partial Listing of Purchased Components for The ADL Model-6 Calorimeter

No.	Item	Model or Type	Supplier
1	High Vacuum Pumping Station	3305	National Research Corp.
2	Vacuum Pump	1402B	The Welch Scientific Co.
3	Oil Circulating Pump	3900 - 3/4 size	Deming Pump Co.
4	Constant Temperature Bath	3E937 - 30 gal	Dayton Co.
5	Heating Elements	TG-130, 3000 w, 240 v	Chromolox
6	Oil Thermostat	Type D5, Model 86, 70°F - 370°F with Copper Thermal Unit, Style B	United Electric Control
7	Dial Thermometers	2281, 50°F - 300°F, 2-1/2" stem	Weston
8	Oil Flow Indicator	63128-12, #2235573, 2 GPM	Fisher-Porter
9	Hydraulic Jack	R618	Black Hawk
10	Ionization Gauge Adaptors	1304	National Research Corp.
11	Thermocouple-Ionization Gauge Control	710B	National Research Corp.
12	2-inch Gate Vacuum Valve	1293	National Research Corp.
13	1-inch Block Type Vacuum Valve	1255	National Research Corp.
14	Thermocouple Selector Switch with Knob, Dial & Index Plate	31-3-0-2 2-13-0-2 48-1-0-9	Leads & Northrup
15	Compound Pressure Gauge	505S-30"-0-15# 2-1/2" dial, 1/4 LM Conn.	U. S. Gauge Co.

No.	Item	Model or Type	Supplier
16	Pressure Gauge 1000 psi	Duragauge 0-1000 amp 733 dial	Ascroft Gauge Co.
17	Dual Manometer	Special	F. W. Dwyer Mfg. Co.

1.8.4 List of Spare Parts

No.	Item	Use	Description
1	O-Ring	Base Plate	12-1/2 I.D. x 1/4 ∅ Neoprene
2	O-Ring	Warm Plate Neck	2" I.D. x 3/16 ∅ Neoprene
3	O-Ring	4" Flange	
4	O-Ring	Sample Chamber	7-1/2 I.D. x 1/16 ∅-Buna-N
5	Stainless Steel Diaphragm	Sample Chamber	9-1/4 O.D. x 0.0015" Thick #304 Stainless Steel
6	Black Paint	Cold and Warm Plate	MMM #9564 Black
7	Rubber Sleeve	Guard Vessel Neck	2" I.D. x 1/4" Thick x 3" Long Natural Rubber Vacuum Hose
8	Ionization Gauge	Bell Jar, Sample Chamber	NRC, Model 507
9	Thermocouple Vacuum Gauge	Same	NRC, Model 501
10	Thermocouple Wire	Warm Plate	#36 (0.005" Dia.) Copper-Constantan
11	Oil	Constant Temperature Bath	300T Pacemaker Turbine Oil, Cities Service Oil Co.
12	Vacuum Oil	Diffusion Pump	#704, Dow Corning Co.
13	Vacuum Oil	Low Boil-Off Meter & Forepump	Super-X, Kinney Pump Co.
14	Bolts	Sample Chamber Neck	5/16-18 NCT x 7/8 Long #304 S.S. Cap Hd.
15	Screws	Warm Plate	#5-40 NCT x 2-1/8 Long Flat Hd.-Brass
16	Bolts	Sample Chamber	3/8-16 NCT x 1-1/2 Long #304 S.S. Cap Hd.
17	Screws	Teflon Bearing	#10-24 NCT x 7/16 Long #304 S.S. Round Hd.

No.	Item	Use	Description
18	Bolts	Bell Jar	1/2-13 NCT x 1-3/4 Long #304 S.S. Cap Hd.
19	Tubing	Manometer Lines & Sleeves	1/4 I.D. Gum Rubber Tubing
20	Manometer Fluid	Manometer	Dwyer, Sp.Gr. 1 #A122

2.0 THE LINE HEAT SOURCE APPARATUS AND THE THERMAL CONDUCTIVITY PROBE

The line heat source apparatus and the thermal conductivity probe represent two experimental variations of the same method for measuring thermal conductivity of homogeneous and heterogeneous materials. In general, the theory of the method can be applied equally well to each apparatus, provided the assumptions used in the analysis apply to each experimental arrangement. The experimental apparatus usually take on different forms. The line heat source is more fragile and is designed primarily for the study of powders under various environmental conditions. The probe is a more rugged instrument suitable for studying the properties of solid, porous, and powdered materials. In the following discussion we will (1) briefly review the historical development of the probe and the line heat source, (2) discuss the theoretical background for the methods, (3) describe the apparatus constructed, tested, and delivered under this study, and (4) provide operating instructions for the apparatus. The similarities and differences between the two approaches are indicated where pertinent to the uses of the instruments.

2.1 HISTORICAL REVIEW

The line heat source method was suggested by Schleiermacher[6] in 1888 and later by Stalhane and Pyk.[7] The first practical use of the method was by Van der Held[8] who used the line heat source for measuring the thermal conductivity of liquids. The line heat source method has also been used for measurement of the properties of insulating materials.[9] We have used the line heat source method at temperatures from -150 to +150°C to measure the thermal conductivity of postulated lunar surface materials.[10] The line heat source method has the advantages that it requires only small quantities of sample, can be used within ultrahigh vacuum chambers, can be arranged so that a powder is deposited on the apparatus after outgassing and other treatments and requires only short times for the experimental measurements.

The first thermal conductivity probe utilizing the principles of the line heat source method was described by Hooper and Lepper.[11] The

principal use of the probe has been in the measurement of the thermal properties of soils.[12,13,14] Other investigations have used the probe for study of insulating materials,[15,16] for rocks and sand packs,[17] and deep sea sediments.[18] We have used the thermal conductivity probe for studies of powder, foam and solid rocks at temperatures from -150 to +500°C.[19,20] The probe method has several of the advantages common to the line heat source method but is better adapted for use with solid and foam materials or where the use of the line heat source is prohibited by its fragile nature.

2.2 THEORETICAL CONSIDERATIONS

2.2.1 The Line Heat Source

The constant heat production by a line source of heat enclosed in an infinite volume of materials produces a cylindrical temperature field. The temperature rise at any point in the material depends upon its thermal conductivity. For a line heat source of strength q per unit length, the temperature, T, at a distance, r, from the source is given by the equation[21]

$$T = -\frac{q}{4\pi k} \; Ei \left[-\frac{r^2}{4\alpha t}\right] \qquad (1)$$

where

$$-Ei\,(-x) = \int_x^\infty \frac{e^{-x}}{x}\, dx \qquad (2)$$

and α is the thermal diffusivity of the material, k is the thermal conductivity, and t is time. The boundary conditions are: $t = 0$, $r \neq 0$, $T = 0$; $t > 0$, $r = \infty$, $T = 0$; and $t > 0$, $r \to 0$, $q = \text{constant} = -2\pi r\, k\, dT/dr$. For small values of $r^2/4\alpha t$, corresponding to small distances from the source, large thermal diffusivities or long time, the exponential integral can be approximated by $+0.5772 + \ln x$; thus, Equation 1 becomes:

$$T = \frac{q}{4\pi k} \left[-0.5772 - \ln \frac{r^2}{4\alpha t}\right] \qquad (3)$$

For temperature rises of T_1 and T_2 at times t_1 and t_2, respectively, Equation 3 gives:

$$T_2 - T_1 = \frac{q}{4\pi k} \ln \frac{t_2}{t_1} \qquad (4)$$

Thus, from the temperature rise at two different times and from the strength of the heat source, the thermal conductivity can be computed. Alternatively, a plot of temperature rise versus logarithm of time should have a constant slope whose value is equal to $q/4\pi k$.

We note that the thermal conductivity values as evaluated by Equation 4 do not depend upon the values of r at which measurements are made, nor on the thermal diffusivity, provided that the values of $r^2/4\alpha t$ are small. This is especially important in experimental work, since it is not necessary to know precisely the location of the temperature measuring device. In many experimental arrangements, it is not possible to wait for small values of $r^2/4\alpha t$ to complete the measurements or the effects of boundaries and heat leaks become large before the values of $r^2/4\alpha t$ are sufficiently small for Equation 4 to be used. In this case, it is still possible to determine values of thermal conductivity (without knowledge of the measurement radius). It can be seen from Equation 1 that the temperature rise at a point in the material is a function only of the heater power per unit length, the thermal conductivity, and a generalized dimensionless time function. Hooper and Chang[14] have presented a monograph for solution of the thermal conductivity in terms of the temperature rise at several times. A graphical method for determining the thermal conductivity from Equation 1 has been used in our previous work[19] and will be discussed later. In most experimental measurements of evacuated powders with the line heat source apparatus, it will be necessary to use a graphical procedure instead of allowing sufficient time for Equation 4 to hold.

The above discussion is based upon measurements using an idealized system; practical considerations result in differences from the above described model and accompanying errors in measurement. The principal sources of error in the line heat source method are: (1) the effects of finite heater wire diameter and length, (2) the effects of the diameter

and length of the temperature sensor, (3) the finite size of the sample, (4) variations in heater power and initial sample temperature, and (5) contact resistance between the sample and the heater wire or temperature sensor. These sources of error have been considered in detail elsewhere.[10] In the design of the line heat source apparatus delivered under this contract, we have considered these sources of error and design criteria so as to reduce measurement error to allowable limits. Conscientious application of the measurement technique suggested later should insure reliable and reproducible data.

2.2.2 The Thermal Conductivity Probe

The theoretical analysis of the thermal conductivity probe has proceeded along two similar methods: (1) an extension of the line heat source theory and (2) analysis of a cylindrical hollow probe. In the first approach, we can consider the probe itself (i.e., the heater portion, insulation, temperature sensor, and sheath) as a line heat source. Equations 1 through 4 will then hold for every point within the material to be tested. In practice it is customary to monitor the temperature of the material at some point near the probe, such as in a sheathed thermocouple attached to the heater sheath.[17] In this case the equations given above hold, and the thermal conductivity of the powder may be found in the same manner as when the line heat source is used. Because the temperature sensor is often located only a small distance from the heater, only relatively short experimental times are required for the term $r^2/4\alpha t$ to be small so that Equation 4 may be used in evaluating thermal conductivity.

In the second approach, equations have been derived for the temperature at the inner radius of a hollow cylinder containing a line heat source.[22] The effects of finite probe dimensions, thermal conductivity, and specific heat are taken into account. The most general equation for the temperature at the internal radius of a hollow probe is given by Blackwell as:[22]

$$T(a,t) = \frac{q}{4\pi k}\left[\ln 4\tau - \gamma + \frac{2k}{bH} + \frac{1}{2\tau}\left\{\ln 4\tau - \gamma + 1 - \frac{\rho_1 c_1}{\rho c}\left(\ln 4\tau - \gamma + \frac{2k}{bH}\right) - \frac{2\alpha}{b^2}(\Delta_1 + \Delta_2)\right\} + 0\left(\frac{1}{\tau^2}\right)\right] \quad (5)$$

where $\tau = \frac{\alpha t}{b^2}$, a and b are the internal and external radii of the probe, H is the "heat transfer coefficient" at the probe wall, $\rho_1 c_1$ and ρc are the volumetric heat capacity of the probe and surrounding material, respectively, γ is Euler's constant, and Δ_1 and Δ_2 are functions of the geometry and thermal properties of the probe only. If the effects of finite thermal conductivity and specific heat of the probe are neglected, Equation 5 reduces to:

$$T = \frac{q}{4\pi k}\left[\ln \frac{4\alpha t}{r^2} - \gamma + \frac{2k}{bH} + \frac{1}{2\tau}(\ln 4\tau - \gamma + 1)\right] \quad (6)$$

For sufficiently large times, i.e., large values of τ, Equation 6 reduces to:

$$T = \frac{q}{4\pi k}\left[\ln \frac{4\alpha t}{r^2} - \gamma + \frac{2k}{bH}\right] \quad (7)$$

It can be seen that Equation 7 is identical to Equation 3, with the exception of the constant term due to the contact resistance at the probe-sample interface. Thus, the simple line heat source theory is valid for the probe for long experimental times. (Note that in Equation 5 all of the terms involving the probe dimensions or specific heats are multiplied by $1/\tau$ so that their influence becomes small for large times.) In experimental practice, probes are generally constructed so that the effects of probe properties are reduced in relatively short times and the probe may be treated as a line heat source. For materials with very low thermal diffusivities it may be necessary to estimate some of the parameters in Equation 6 or 7 and use them in an iterative technique to determine the thermal conductivity.

The errors encountered in the probe method are similar to those accompanying the line heat source method; of principal importance are finite

length and diameter of the probe, contact resistance and sample size. Several investigators have discussed these errors[12,23] and studies are now in progress in which these errors are being evaluated more fully.[24] Examination of the errors indicates that for small experimental times, the probe test results deviate from those predicted by line source theory because of the finite dimensions of the probe and the contact resistance. At very long times, the probe test results deviate from those predicted from theory because of axial heat losses and the finite sample size. Between the short and long time portions where deviations occur, the probe test results should be predictable by Equation 1 or 4. It is desirable to have the valid time portion occur at relatively short experimental times and under conditions such that the logarithmic formulation (Equation 4) can be used. In the design of the probe, we have taken some of these factors into account; however, the ultimate suitability of the probe, the experimental times and the measurement errors will depend to some degree on the properties of the material being studied.

2.3 DESCRIPTION OF APPARATUS

2.3.1 Line Heat Source Apparatus

The line heat source apparatus consists of parallel heater and thermocouple wires, a support structure, and a sample holder. Figure 24 shows the support structure and wires fixed within the sample holder. Figure 25 shows the support structure and wires of an almost identical but slightly longer apparatus. The support structure for the heater and thermocouple wires consists of a stainless steel base, 1" x 6" x 1/8". Four Pyrex glass supports have been attached with epoxy resin to 1/16" holes drilled in the stainless steel base. The supports are hook shaped and extend outward toward the edges of the base. Four ceramic-metal insulating seals are attached to the steel base approximately 1/2" from the glass supports.

A 0.001" diameter constantan heater wire is attached to one pair of glass supports with epoxy resin. The wire is firmly attached so as to be taut without undue strain. The wire continues from the glass supports and is soldered to the ceramic-metal binding posts. The total heater wire

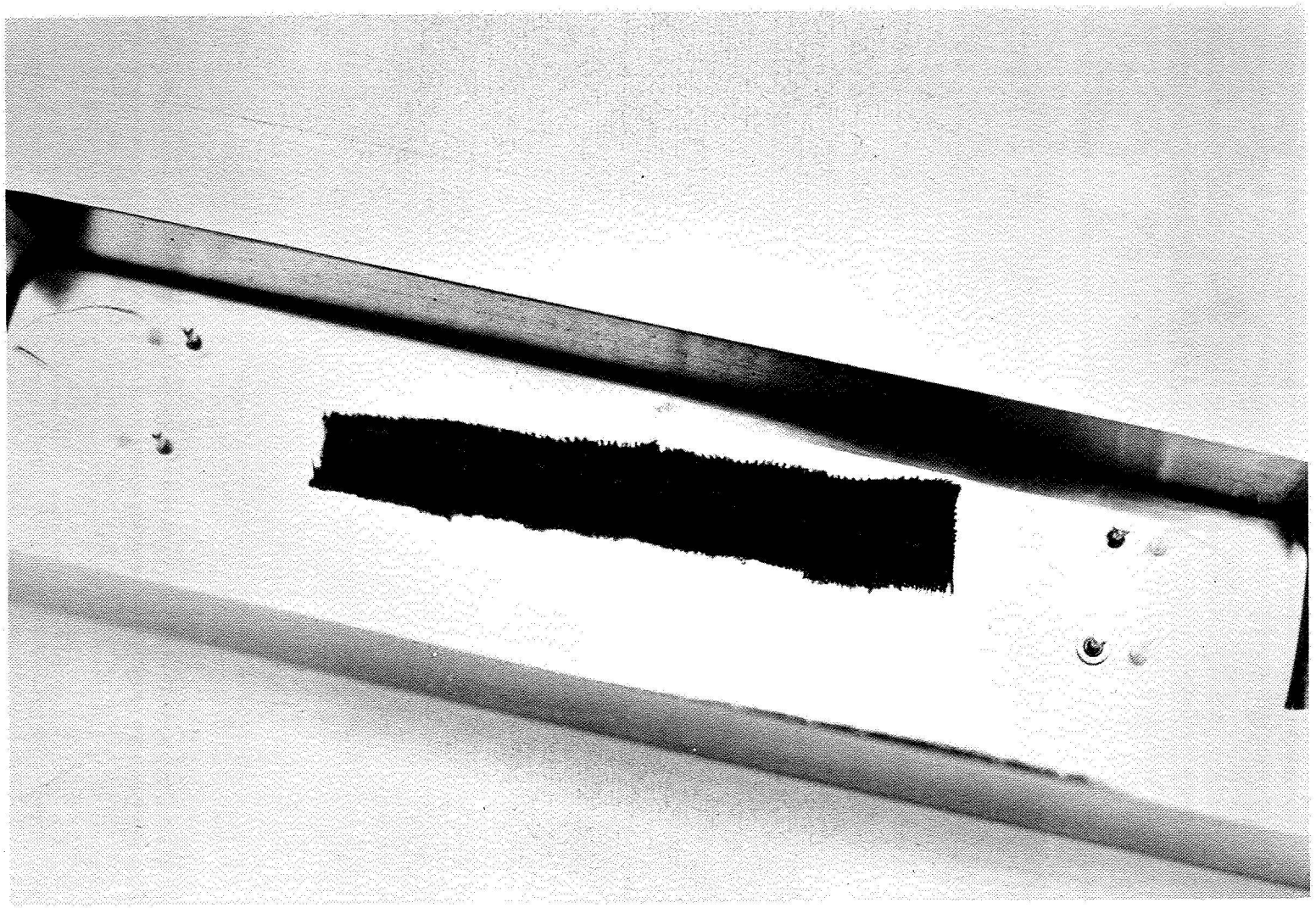


FIGURE 24 LINE HEAT SOURCE APPARATUS IN THE SAMPLE HOLDER

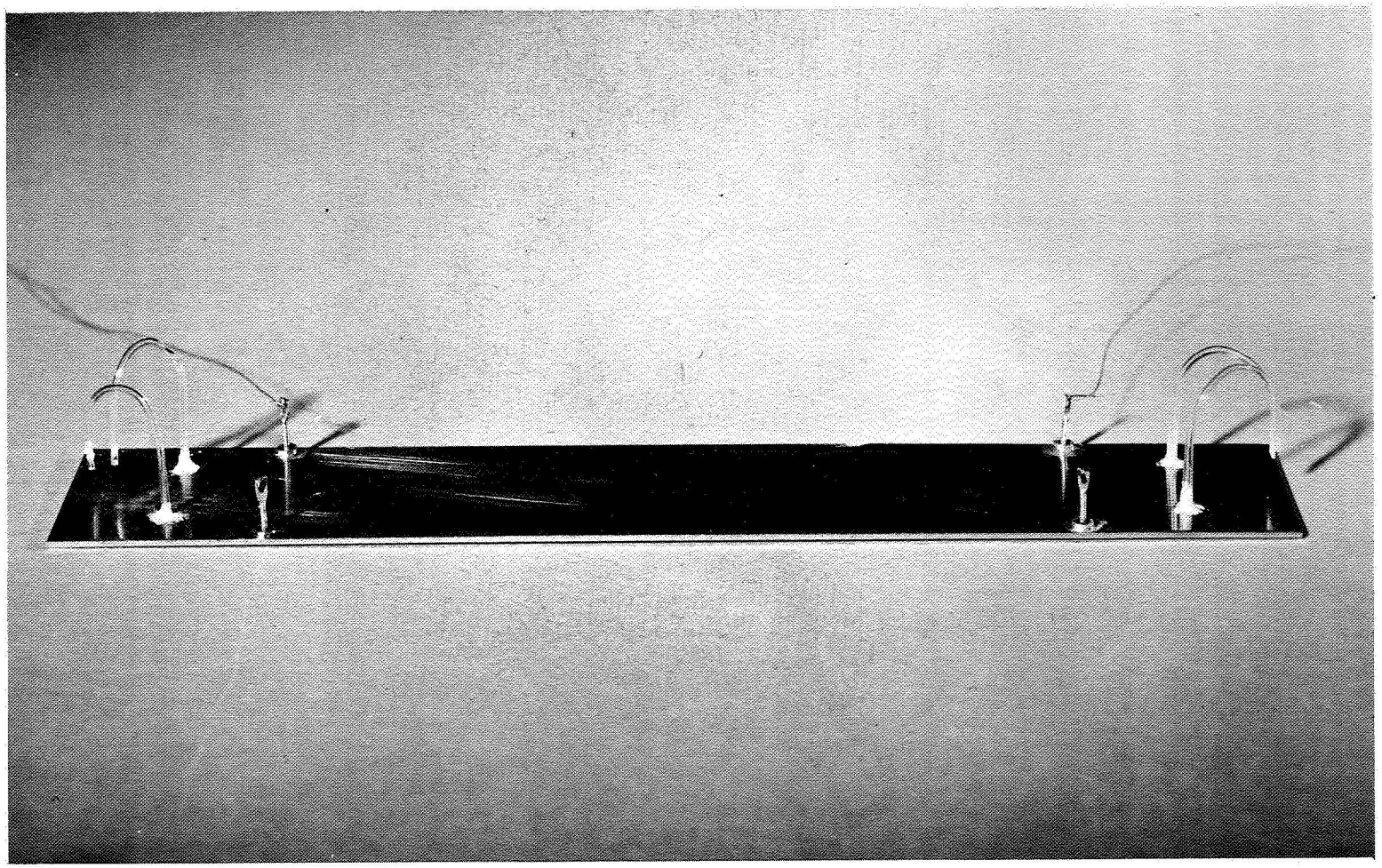


FIGURE 25 LINE HEAT SOURCE APPARATUS

length is 8 inches; the supported length approximately 5-1/2", and each end loop (between the glass supports and the binding posts) is approximately 1-1/8" long. A 0.001" diameter iron-constantan thermocouple is placed parallel to and spaced approximately 3/64" from the heater wire and is supported by the second set of glass posts. Both wires are approximately 1/4" above the steel base. The "end loops" of the thermocouple wires are approximately 1-1/8" long, the wire length between the glass supports is approximately 5-1/2". The iron-constantan thermocouple junction was made by placing the two wires next to each other, silver soldering the junction, and trimming off the excess wire. Heater wire leads 0.015" stranded copper wire, approximately 18" long are attached to the binding posts. Heavier No. 36 thermocouple lead wires, about 36" long, were attached to the binding posts. The total electrical resistance of the heater and leads is 177.75 ohms. The resistance of the lead wires is less than 1% of the total resistance. An iron-constantan thermocouple was chosen to reduce axial heat losses (iron has a thermal conductivity less than 1/6 of copper). The length and diameter of the heater and thermocouple wires are chosen on the basis of previous experience with the line heat source apparatus in which it was shown that axial heat flow and other errors are substantially reduced using this configuration.[10] Although the wires are positioned firmly, they are quite fragile. They should never be handled directly, samples should be deposited on them with care, and the apparatus should be kept in a moisture-free environment.

The sample holder for the line heat source is shown in Figure 24 and an engineering drawing is given in Figure 26. The holder was machined from a copper block; holes were bored along the walls and base for cooling passages. The cooling passages were connected and 1/4" copper tubing was attached to the entrance and exit ports of the holder. A cooling or heating fluid can be passed through the walls and base of the holder to bring the samples to the desired temperature. The sample holder was nickel plated, then chromium flashed, to seal and maintain the condition of the copper and to reduce outgassing. The ends of the copper tubes can be connected by flanges to "pant legs" for the feedthroughs of the base

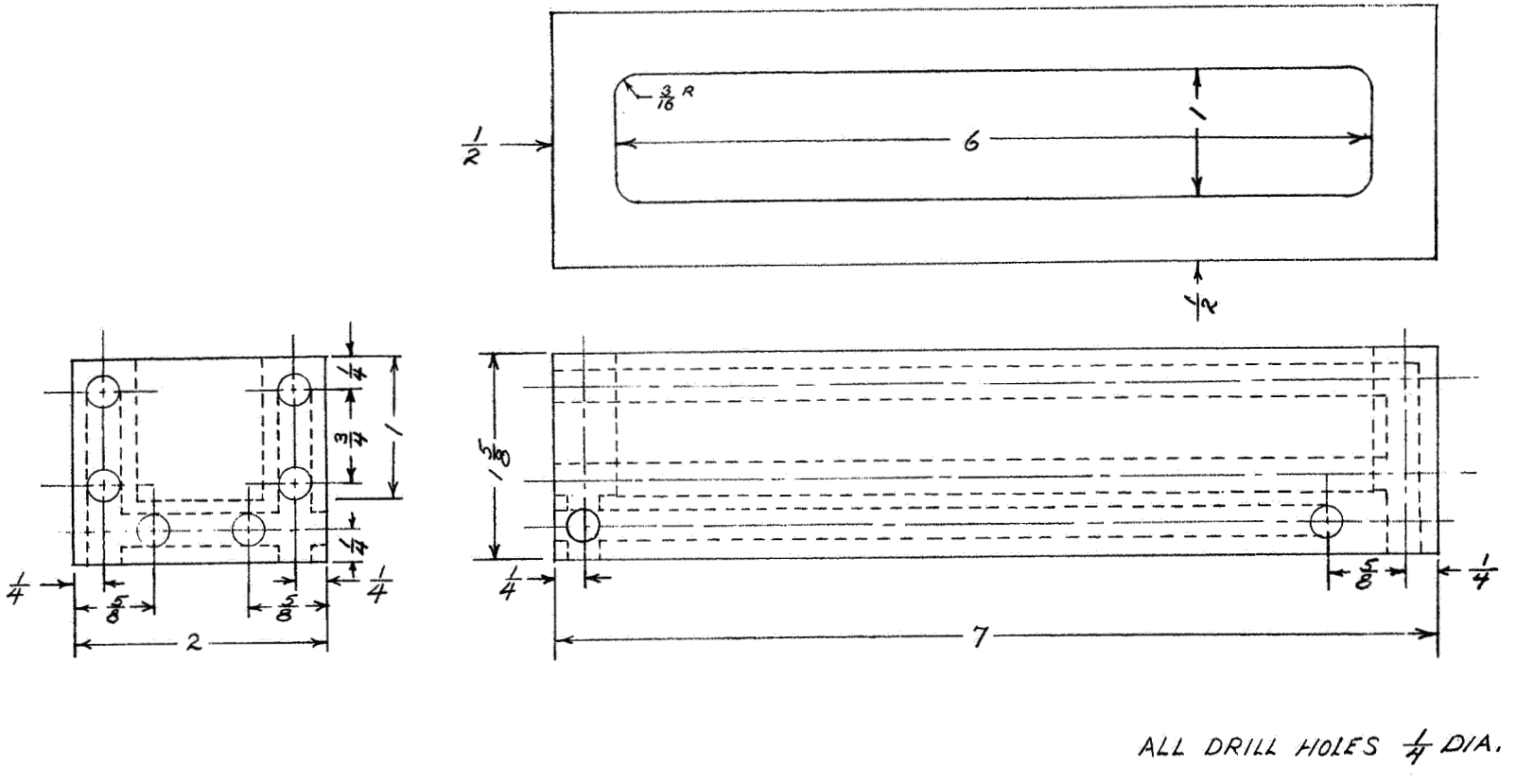


FIGURE 26 CONSTANT TEMPERATURE LINE SOURCE HOLDER

plate and bell jar assembly described later. For use in the high vacuum chamber, the copper tubes can be attached directly to high vacuum feed-throughs. The stainless steel base holding the heater and thermocouple wires can be attached to the sample holder by two screws.

2.3.2 Thermal Conductivity Probe Apparatus

The thermal conductivity probe is shown schematically in Figure 27. The bifilar coil is wound with 0.0031" diameter Teflon-coated constantan wire. The resistance of the heater wire and leads is 213.96 ohms. The coil length is approximately 4-13/16 inches. A 0.0031" diameter Teflon-coated copper-constantan thermocouple (welded joint) is inserted within the coil and at its midpoint. The probe is filled with epoxy resin (Epon 828, DMP-30, and methyl Nadic anhydride mixture) which was cured at approximately 100°C. The outer sheath of the probe is type 304 stainless steel 0.049" O.D., 0.037" I.D. The top of the probe consists of a cone-shaped holder in which the smaller heater wires are connected to copper leads. The thermocouple leads were continuous for several feet of length. The bottom and the top of the probe are filled with Toroseal, a low vapor pressure epoxy resin manufactured by Varian Associates. The heated length of the probe (for use in calculations) is 4-13/16 inches. Filling the probe with epoxy resin prevents possible outgassing due to leaks in the probe sheath and therefore eliminates the possibility of internal contact resistances within the probe. Use of Toroseal for the ends also reduced outgassing of the probe. This type of probe has been used in our previous studies and found to give satisfactory results in both atmospheric pressure and vacuum environments.

The probe sample holder consists of a copper sheet formed into a cylinder 4-3/4" I.D. and 6-5/8" high. One-quarter inch copper tubing has been silver soldered to the wall and bottom of the sample container; legs have been provided to maintain clearance between the bottom of the sample container and the base plate. A copper lid can be attached to the holder by wing nuts. The copper tubing can be attached to pant legs or other means for extending to the outside a vacuum chamber. The sample holder has been nickel plated and given a dull chromium finish to reduce outgassing.

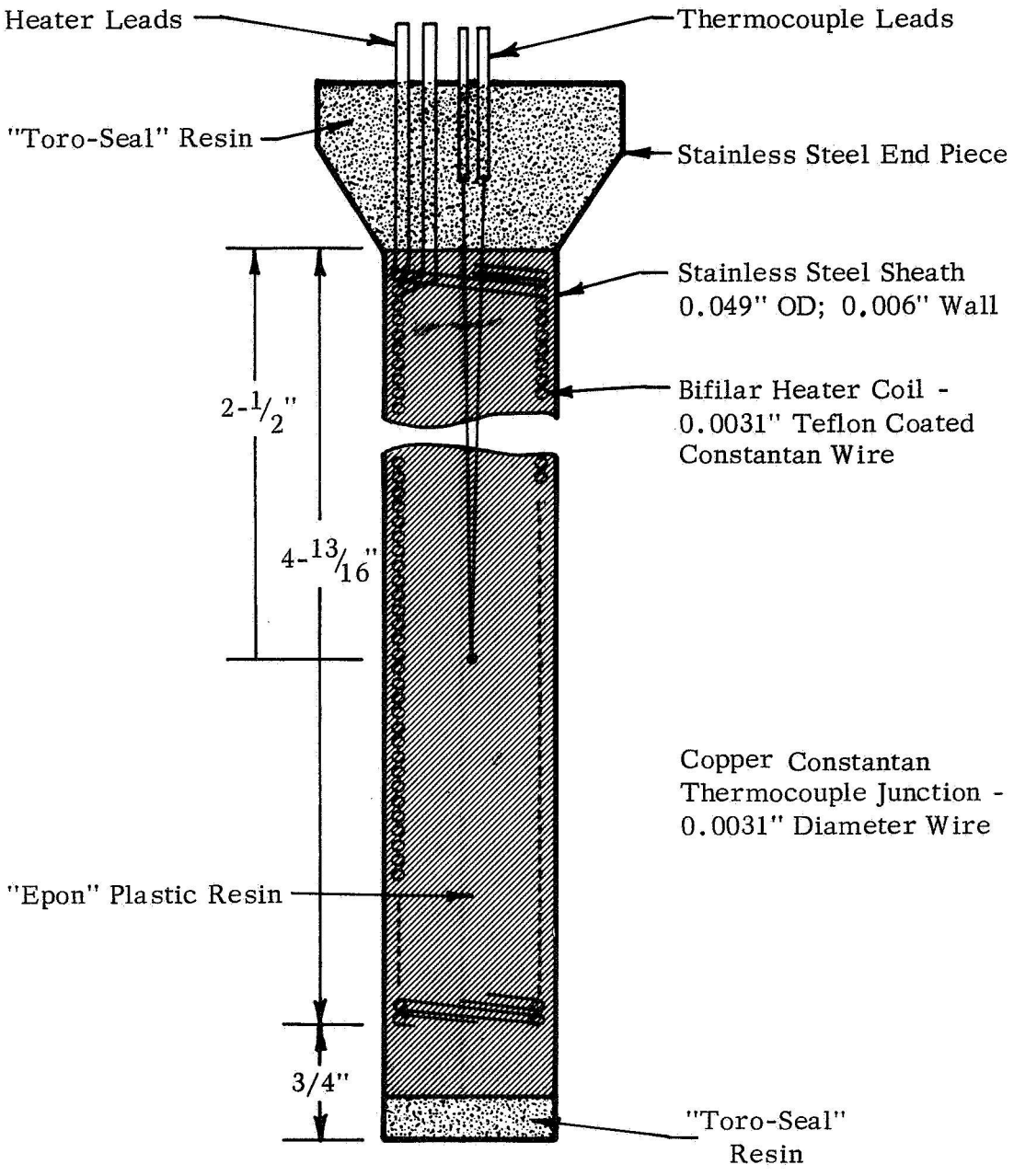


FIGURE 27 SCHEMATIC DIAGRAM OF THERMAL CONDUCTIVITY PROBE

Holes in the container provide passages for evacuation. Figure 28 is a photograph of the probe and the sample holder. A small brass screen cup, approximately two inches diameter has been provided for holding powders where sample volumes are limited. The space between the screen cup and the copper sample holder may be filled with aluminum beads (or other material) to provide good thermal contact between the sample and the temperature controlled copper container. The copper tubing leads should be attached to a thermostatically controlled oil bath or other heating or cooling system.

2.3.3 Base Plate and Bell Jar Assembly

A base plate and bell jar assembly were supplied as the environmental chamber for line heat source and probe measurements. A steel base plate, approximately 14" diameter with a 4" port drilled in the center, was used. An elbow with a 4" flange was welded to the base plate so that it could be attached to the 4" pumping system provided with the cold plate apparatus. A standard 12" diameter Pyrex bell jar with a wire mesh shield was also supplied. In addition to the main pumping port, the base plate contained three vacuum seals: (1) a Conax type TG-20-A-6 six-wire feedthrough for the heater and thermocouple wires of the line heat source and probe and (2) two compression type seals (NRC 1313) for the pant legs of the circulation system. The base plate assembly is shown schematically in Figure 29. The base plate assembly was nickel plated, chromium flashed and leak checked using the 4" pumping system.

2.4 INSTRUMENTATION

Instrumentation for the line heat source and thermal conductivity probe apparatus was not supplied as part of this contract. The instrumentation available at the Research Projects Laboratory, when properly connected and the ground loops eliminated, should be adequate for the measurements.

The instruments required for measurement with either the line heat source or probe include: a regulated d.c. power supply, a K-3 potentiometer with standard cell and auxiliary battery, a galvanometer or nullmeter for use with the K-3 potentiometer, a double pole-double throw knife switch

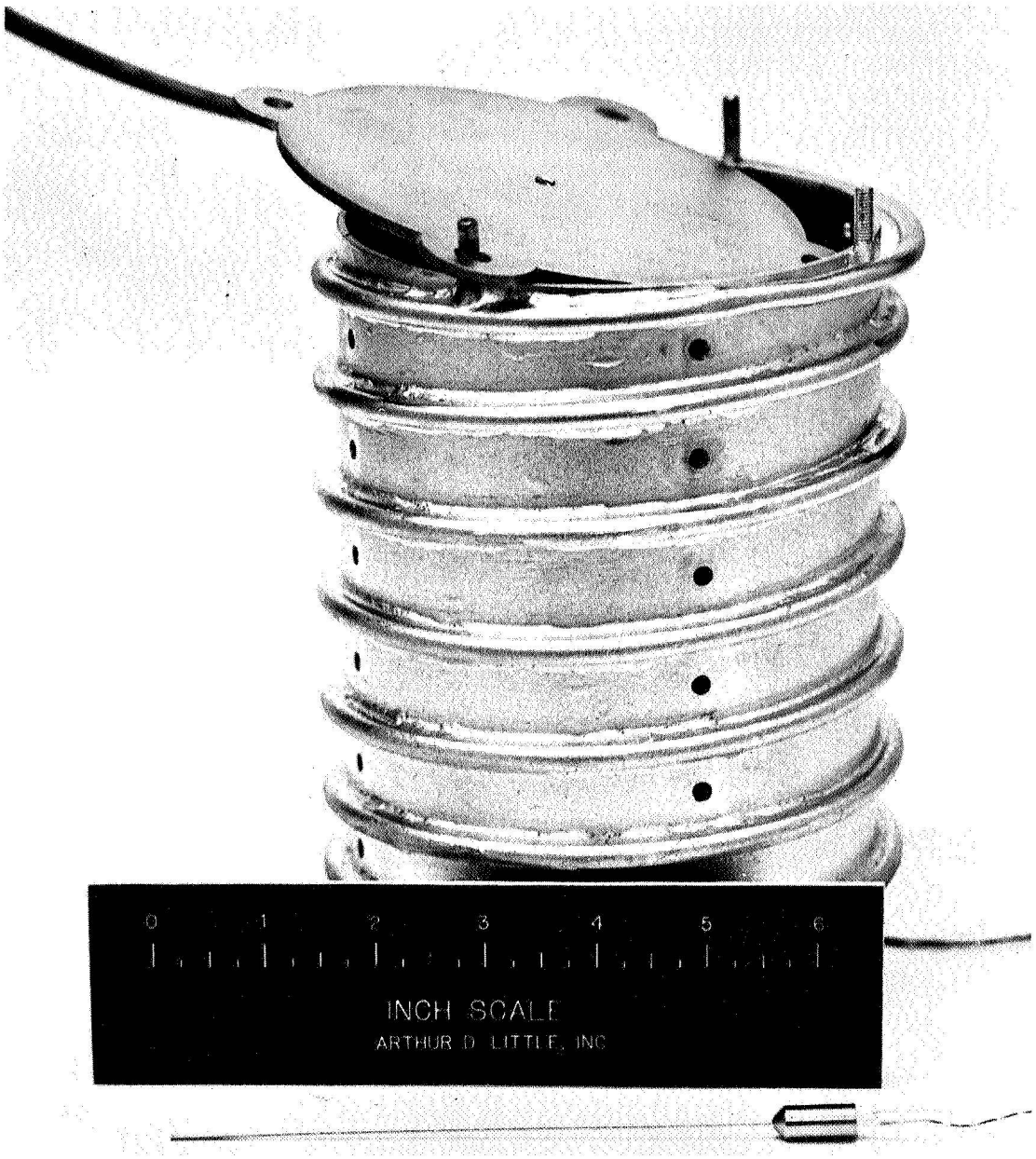
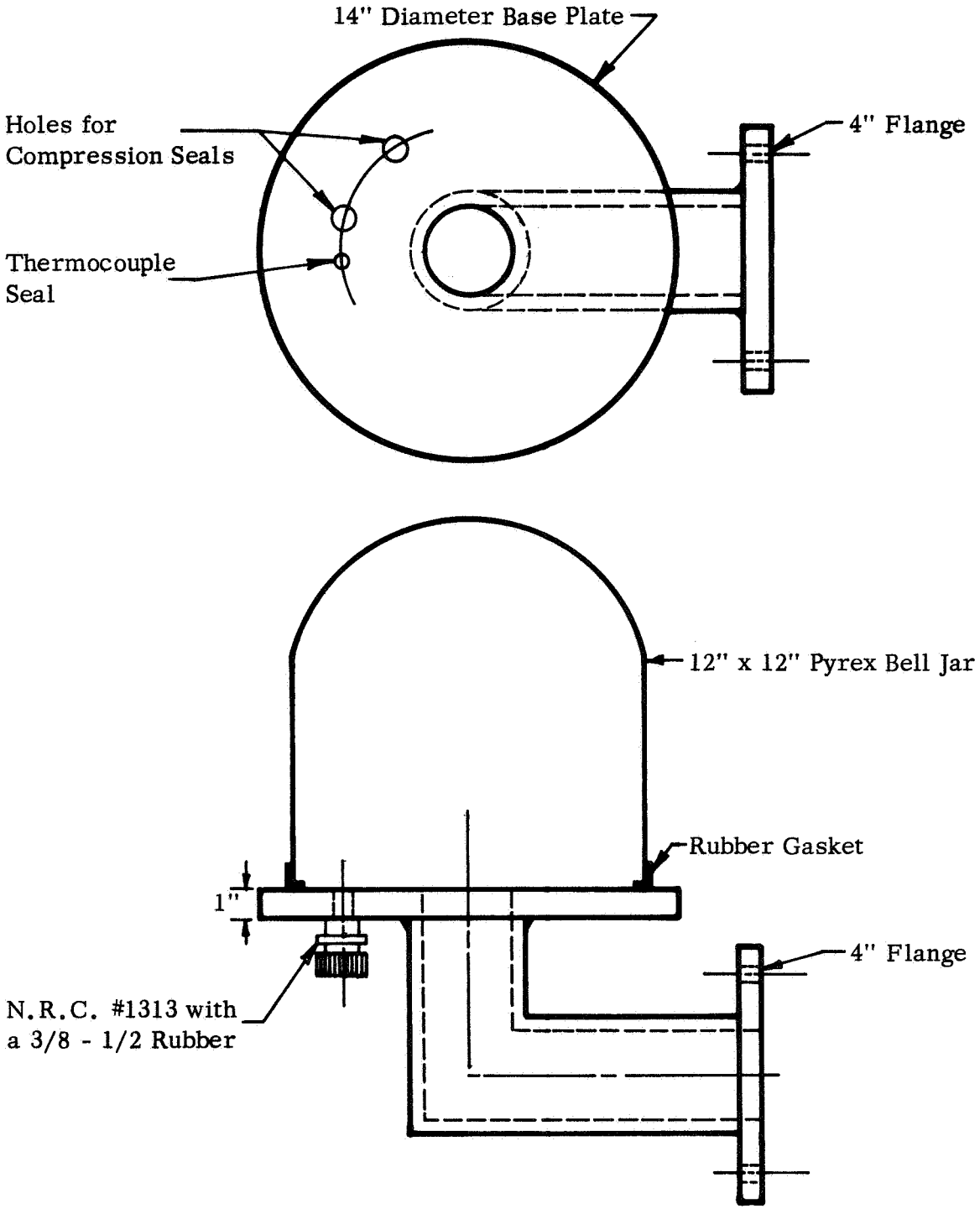


FIGURE 28 THERMAL CONDUCTIVITY PROBE AND SAMPLE HOLDER



Note: Not Shown-Conax #TG-20-A-6 Fitting for Power & T.C. Wires.

FIGURE 29 SCHEMATIC DIAGRAM OF BASE PLATE ASSEMBLY

(a single pole-double throw switch may be used with an alternate wiring system), a microvolt amplifier and recorder (a combination that will give full-scale chart reading of 100 microvolts will be desirable), an accurate voltmeter and ammeter, a thermocouple reference junction, and a timer to check the chart speed. In addition, a decade resistor box with a capacity of 1000 ohms (0.1 to 1000) will assist in setting the power supply. Figure 30 shows the general instrumentation diagram for hooking up either the probe or the line heat source. Figure 31 shows the method for attaching the thermocouple circuit to the potentiometer and recorder-amplifier.

A principal problem in the instrumentation is reduction of system noise and elimination of the effects of ground loops. There are no specific rules for reducing these effects, however, general good instrumentation practice should suffice. The heater and thermocouple wires should be shielded, any thermocouple junctions should be kept at constant temperature and insulated from air currents, the recorder and amplifier should be shielded from any high magnetic or electric fields. Depending upon the type of instruments (recorder and amplifier) available and the impedances of the thermocouple circuit, modifications of the components may be required to increase the sensitivity and time response of the recording system. A competent instrumentation engineer should be capable of improving the circuit to the point where noise is kept below one microvolt and the recorder response is sufficiently rapid to achieve accurate temperature rise measurements.

During the installation of the apparatus, instrumentation available at the Research Projects Laboratory was set up for demonstration tests. This set-up is typical of that required for accurate measurements; reduction of noise and the effects of ground loops and improving the impedance match of the components will improve the quality of the experimental data.

2.5 OPERATING INSTRUCTIONS

2.5.1 Line Heat Source Apparatus

The following instructions describe the procedure to be followed in the normal operation of the line heat source.

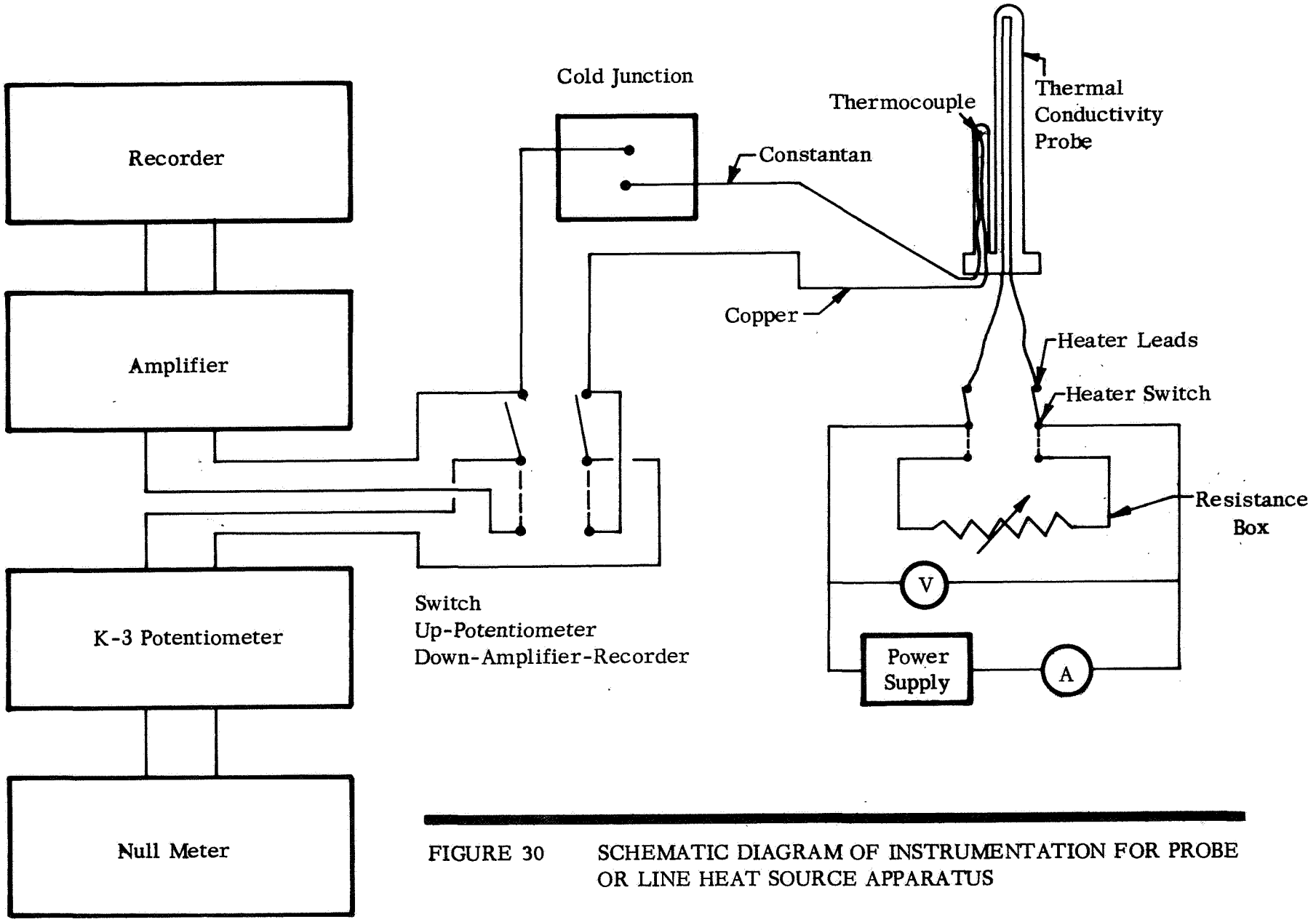


FIGURE 30 SCHEMATIC DIAGRAM OF INSTRUMENTATION FOR PROBE OR LINE HEAT SOURCE APPARATUS

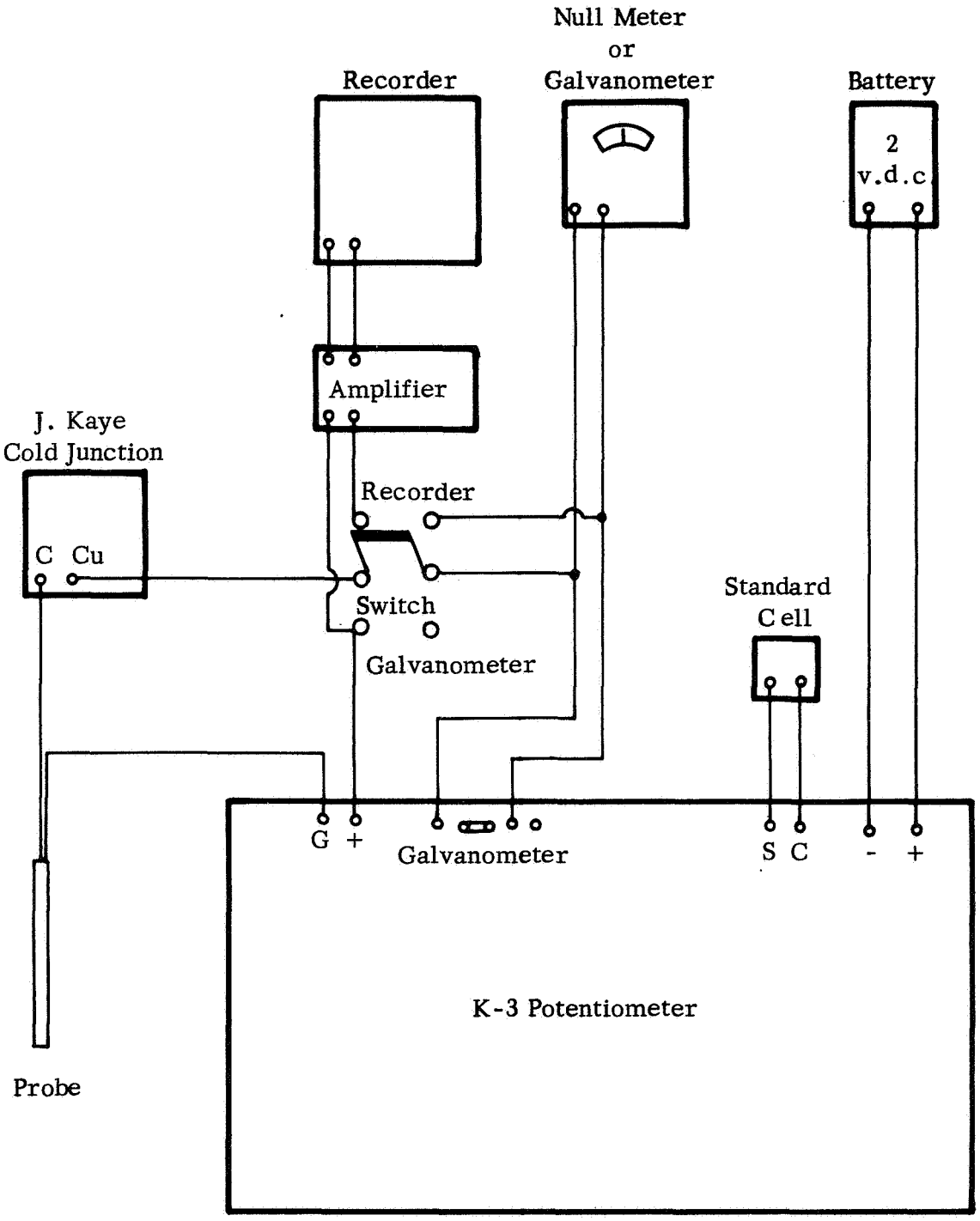


FIGURE 31 THERMOCOUPLE WIRING DIAGRAM FOR PROBE OR LINE HEAT SOURCE

A. Pretest Checkout

1. Prior to testing examine the line heat source apparatus to insure good condition and taut position of the wires.

2. Check the electrical continuity of the thermocouple and heater wire.

3. Check sample holder to remove any foreign material and check fluid passages to insure the absence of leaks.

B. Equipment Assembly and Sample Loading

1. Attach the base plate assembly to the pumping port of the vacuum system.

2. Place the line heat source sample holder on the base plate assembly (or other location to be used).

3. Attach the circulating fluid tubes to the couplings in the "pant leg" seal assemblies; attach the other ends of the fluid feed tubes to the desired temperature controlled bath (e.g., cold water tap, auxiliary oil bath ports on calorimeter). Change washers, flare fittings and rubber gromets as required to insure tight connections.

4. Carefully position the line heat source base and wires in the sample holder with the screws provided. Attach the iron and constantan thermocouple wires and copper heater leads to the appropriate terminals in the Conax 6-wire feedthrough (soft solder or good mechanical joints will suffice under most conditions). Tighten Conax fitting if required.

5. Check to insure that there is no electrical connection between the heater or thermocouple leads and the base plate or sample holder.

6. Carefully fill the sample holder with the sample of the desired size and density. No voids should be formed in the holder and the heater and thermocouple should be covered as uniformly as possible. The wires should be covered with at least 3/8" of sample. If sufficient sample is not available, cover only the central 3-inch portion of the apparatus with the material to be measured and cover the outer portions with a similar powder.

7. Place the bell jar and rubber gasket on the base plate assembly using the appropriate sealant.

C. Establishment of Sample Environment

1. The sample environment should be established at the desired conditions of gas pressure and temperature by means of control of the vacuum system and the temperature bath. Initial pumpdown (in the range from atmospheric to several torr) should be slow to avoid movement of the sample.

2. When the desired gas pressure and sample temperature are reached, as measured by the vacuum gauges and the thermocouple, the sample should be allowed to remain until pressure and temperature equilibrium is reached. This will normally be from 1/2 hour at atmospheric pressure to 12 hours or longer at reduced pressures.

D. Checkout of Instrumentation (To be carried out after A and B)

1. Check all recorder, potentiometer, thermocouple and heater wire connections.

2. Standardize the K-3 potentiometer. Check span on recorder and time response and noise level of amplifier-recorder system. Establish chart speed of recorder. Check operation of reference junction.

3. Test proper functioning of power supply, volt and ammeters, and switches but do not apply a voltage to the heater wire.

E. Thermal Conductivity Tests

1. Measure initial temperature of the sample (after the system has come to thermal equilibrium) using K-3 potentiometer and nullmeter or galvanometer. (K-3 should be standardized periodically.)

2. Set decade resistance box at a value corresponding to the resistance of the line heat source; set the power supply so that the voltage will be applied to the external resistor (not the line source heater wire). Turn on the power supply, adjusting the current and voltage to levels which will give a desired temperature rise in the sample. (In general, the voltage and current levels will be determined by previous experience. It is normally desired to have temperature rises of 2-10^{o}C--as measured by the thermocouple--during the 5 to 60 minute test period. Estimates of the power required can be made by using Equation 1 with

appropriate assumed values of thermal properties and geometrical factors. When in doubt as to the power to use, start with very small applied voltages and increase them until the desired temperature rises are obtained. Tests under vacuum conditions should always be carried out at lower power levels than tests at atmospheric pressure.)

3. Allow power supply to come to steady operating conditions. Turn on amplifier and recorder and allow to warm up.

4. Recheck and record the temperature reading of thermocouple. The thermocouple switch should be in position to read temperature directly. Start the recorder chart drive (speeds of 1" per minute are adequate for most tests), move the thermocouple switch to the recorder position. Observe the temperature trace on chart--the temperature should not change by more than $\pm$ 3 microvolts over a 10-minute period.

5. When temperature is steady, reset recorder pen trace to zero by using the K-3 potentiometer.

6. When conditions are again steady, move the power switch so that the voltage is applied to the line heat source. Indicate on the recorder chart the location at which the power is turned on. When the recorder pen reaches full scale deflection, reset the pen to zero using the step switch on K-3 potentiometer (or other predetermined movement of vernier scale on K-3).

7. Record the voltage applied to the heater wire and the heater wire current. Check the value several times during the test.

8. Observe the recorder chart to note any abrupt changes. Record sample pressure, heating or cooling fluid temperatures, recorder chart speed and other pertinent identification data.

9. Continue test for sufficiently long duration so that the rate of rise of temperature is very small--durations of 10 minutes for unevacuated materials and 60 to 80 minutes for evacuated materials should be sufficient.

10. Shut off power to heater wire. The cooling of the sample, for a time equivalent to the heated time, may be recorded and used to check the heating data. This is not essential to the calculations or measurement.

11. Allow the sample to come to temperature and pressure equilibrium before repeating tests.

F. Data Reduction

1. The data on the chart recorder gives the temperature rise (in terms of thermocouple output) as a function of time. This data should be converted to a temperature rise-time form. This can be accomplished from the known or calibrated output of the iron-constantan thermocouple. For approximate results, the handbook values of emf versus temperature for iron-constantan thermojunctions should be used. For more accurate work, the thermocouple junction should be calibrated (prior to tests) to determine its output in the temperature range of the measurement. A Beckman thermometer or a vapor pressure thermometer is useful to establish the calibration. It should be remembered that the absolute temperature level is not important in the calculations; the temperature rise with time above the initial value is the important value.

2. Having established the relationship between temperature and time, a plot of temperature rise versus logarithm of time should be made. For samples at relatively high thermal conductivity, this type of plot will be linear for a considerable time portion, with departures from linearity at very small times (less than 1/2 minute) and at longer times (greater than 5 to 10 minutes). For samples with low conductivity a linear curve will probably not be observed, except at large experimental times. If it is established that the curve is linear for a substantial portion, draw the best straight line through the curve.

3. The heater power per unit length can be determined from the product of the current times voltage, corrected for any resistance in the lead wires, divided by the given total heater wire length.

4. From the straight line portion of the temperature-logarithm time response, the thermal conductivity may be calculated from Equation 4 using any corresponding times and temperature rises.

5. If a linear relationship is not clearly established, the experimental data should be plotted as logarithm of temperature versus logarithm time. The experimental curve should be "matched" with a

previously constructed plot (on the same scale coordinate paper) of ln -Ei(-1/x) versus ln 1/x. It is necessary to keep the coordinate axes of the graphs parallel during the matching. The curves are matched when the greatest segment of the experimental data coincides with the theoretical curve. When the curves are matched, determine the temperature rise which corresponds to a value of -Ei (-1/x) of unity and call this temperature T*. The thermal conductivity is obtained from the equation:

$$k = \frac{q}{4\pi} \frac{1}{T^*}$$

where q is the heater power per unit length as determined in step 3. The curve matching should be tried several times to check on the value of T*.

6. Experience will determine which method gives the most reliable thermal conductivity results. In general, samples with low thermal conductivities will require the "curve matching method". The line heat source apparatus should work best with evacuated samples with low thermal conductivities rather than with samples at atmospheric pressure.

2.5.2 Thermal Conductivity Probe Apparatus

The operating instructions for the probe are similar to those for the line heat source apparatus.

A. Pretest Checkout

1. Prior to test, examine the probe to determine any apparent breaks in the lead wires or to remove any foreign material.

2. Check the electrical continuity of the heater and thermocouple wires. There should be no electrical continuity between the probe sheath and any of the probe lead wires.

3. Check sample holder to insure against leaks.

B. Equipment Assembly and Sample Loading

1. Place the sample holder on the base plate assembly. Attach the fluid flow tubes in a manner similar to that described for the line heat source.

2. Attach the heater and copper-constantan leads to the appropriate terminals in the Conax 6-wire feedthrough as described earlier.

3. Check to insure that there is no electrical continuity between the heater and thermocouple leads and the base plate.

4. Place the sample in the sample holder. Use the brass screen and aluminum beads if sufficient sample is not available. Place the sample chamber lid over the probe or probe lead wires.

5. Carefully insert the probe into the center of the sample (a sample radius of at least 1" around the probe should be provided). For a powder material it is sufficient to gently push the probe into the sample. For a rigid sample it is necessary to drill a hole in the sample, slightly smaller than the probe and then insert the probe as a press fit into the hole. An alternate technique is to drill a slightly oversized hole (e.g., 0.010 to 0.020" larger), fill the hole with mercury or other high conductivity contacting material, and place the probe in the hole, displacing some of the contact fluid. The narrow portion of the probe should be fully inserted in the sample. Do not use mercury as a contact agent if tests are to be conducted in vacuum.

6. Close the sample container and place the bell jar and gasket on the base plate.

C. Establishment of Sample Environment

The sample environment is established in the same manner as described for the line heat source apparatus.

D. Checkout of Instrumentation

The checkout of instrumentation is the same as described above for the line heat source apparatus.

E. Thermal Conductivity Tests

The test procedure using the probe apparatus is identical with that of the line heat source with several exceptions.

1. The resistance box should be set at a value corresponding to the probe resistance.

2. The heater power required to obtain desired temperature rises can be estimated from previous experience or from Equation 5 with appropriate values of thermal properties and probe dimensions. The higher the thermal conductivity of the sample, the higher the power to be applied to the heater to obtain a 2-10°C rise during the test time.

3. The test duration for the probe will generally be less than for the line heat source for the same material. For higher conductivity samples such as solid or porous rocks at atmospheric pressure experimental times of only about 5-8 minutes are required. For low conductivity materials, 30-50 minutes should be adequate.

F. Data Reduction

Data reduction methods for the thermal conductivity probe are identical to those of the line heat source with the following exceptions and comments.

1. The handbook value for the copper-constantan thermocouple emf-temperature relationship should be used or the thermocouple should be calibrated in the temperature region of interest.

2. Except for samples with very low thermal conductivity, it will normally be possible to obtain a linear relationship between temperature and logarithm time so that the thermal conductivity can be obtained from Equation 4. For materials with higher thermal conductivities, such as porous or solid rocks, the linear portion of the test will usually occur between 1/2 and 5 minutes.

3. The heated length of the probe, used in calculations, is 4-13/16 inches (12.2 cm).

2.6 SPARE AND REPLACEMENT PARTS

Replacement parts should not be needed for the line heat source apparatus and thermal conductivity apparatus themselves. The base plate assembly and sample holders may require the following:

1. Fluid Tubing Fittings - Copper flare washers for 1/4" tubing #625-F Imperial Brass Co., Chicago, Illinois.
2. Compression Seals - National Research Corporation, Cambridge, Mass., #1313 with 3/8" - 1/2" rubber seal.

3. Bell Jar and Gasket - National Research Corporation, Cambridge, Mass., 12" x 12" Pyrex Bell Jar #3191-21378, Gasket #3949.

4. Thermocouple Feedthrough - Conax Corporation, Buffalo, New York, Type TG-20-A-6 with six-wire rubber bushing.

2.7 SAMPLE CALCULATIONS

2.7.1 Thermal Conductivity Probe

This sample calculation is given for Test 1 using the thermal conductivity probe operated on January 20 at the Research Projects Laboratory.

Sample Material - Pumice Powder

Gas Pressure - Atmospheric (assumed 760 torr)

A. Estimate of Heater Power Requirements

At atmospheric pressure, experience shows that a power of 5mw/cm is desirable for probe measurements with pumice powder.

Probe heater length = 4-13/16" = 12.22 cm

Required power = 5mw/cm x 12.22 cm = 61.1 mw

Probe resistance = 214 ohms

Power = V^2/R; $V = \sqrt{214 \times 0.0611}$ = 3.6 volts

Approximate heater voltage should be 3-4 volts.

B. Reduction of Temperature Data

Initial temperature = 1.012 mv = 25.6°C

In this temperature range, the output of the copper-constantan junction is 0.0408 mv/°C (handbook value). Using this value we prepare a table of temperature rise versus time as follows:

Time (min)	Chart Reading (microvolts)	Temperature Rise (°C)
0.25	45	1.10
0.50	56	1.37
0.75	62.5	1.53
1.0	67.5	1.65
1.5	74.0	1.81
2.0	79.0	1.94
2.5	82.0	2.01
3.0	84.5	2.07
3.5	87.0	2.13
4.0	89.0	2.18
4.5	90.5	2.22
5.0	92.0	2.26
5.5	93.2	2.28
6.0	94.5	2.32
6.5	95.4	2.34
7.0	96.3	2.36
7.5	97.4	2.38

A plot of the temperature rise versus time is shown in Figure 32. Note that a linear relation is observed from less than 1 minute to about 6 minutes. The straight line was drawn by inspection through the points.

C. Calculation of Thermal Conductivity

For this test, the following data was obtained:

V = 3.89 volts

I = 18.2 mamps

Power = I x V = 70.9 mw

Power/Length = 70.9 mw/12.22 cm = 5.80 mw/cm

Assume: t_1 = 0.5 min

t_2 = 5.0 min

Then from the plot of temperature rise versus time, we obtain

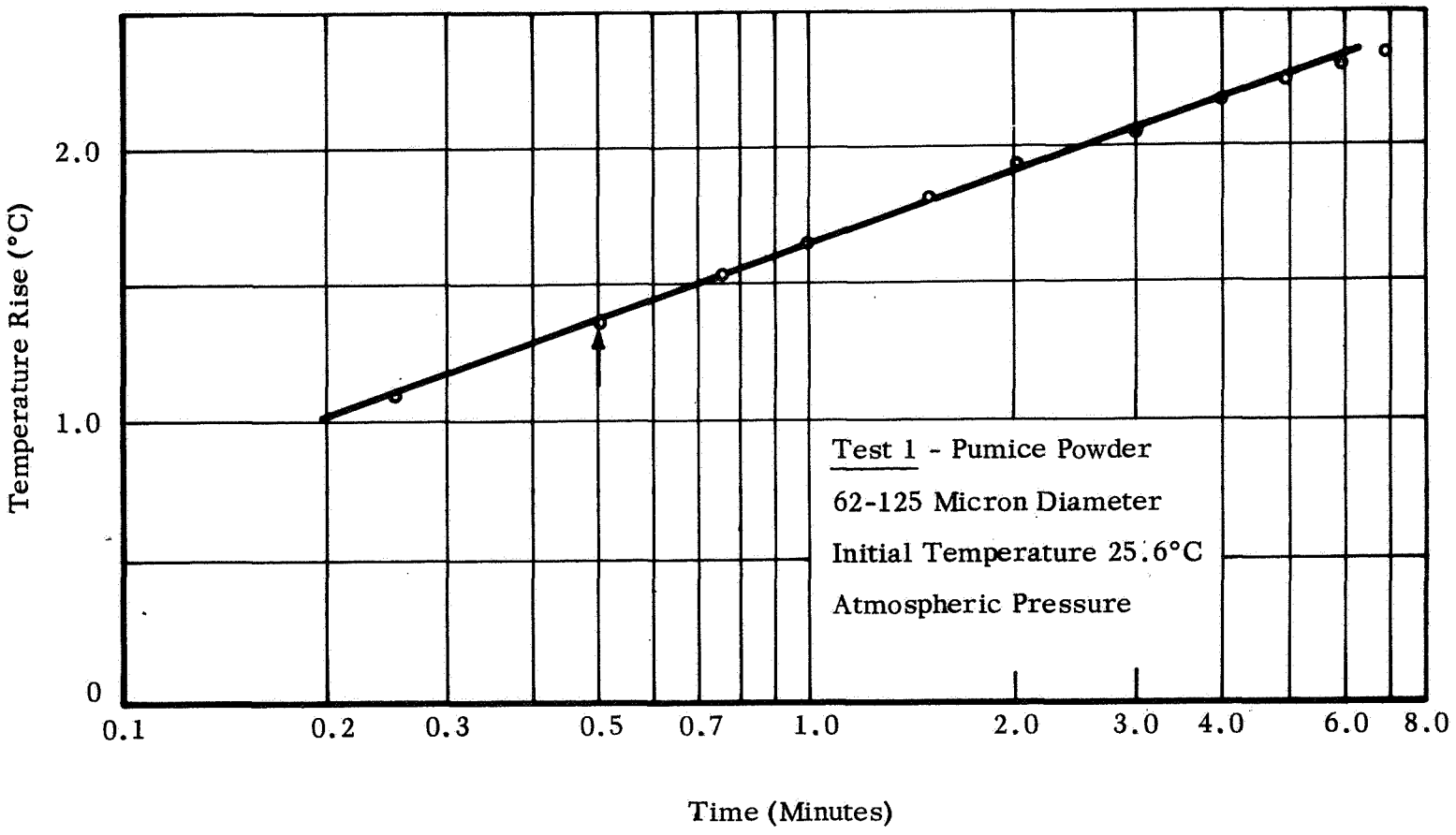


FIGURE 32 TYPICAL DATA FROM THERMAL CONDUCTIVITY PROBE

$T_1 = 1.38^{\circ}C$

$T_2 = 2.27^{\circ}C$

Using Equation 4 we obtain

$$k = \frac{q}{4\pi(T_2 - T_1)} \ln t_2/t_1$$

$$k = \frac{5.80 \times 10^{-3} \times 2.303}{4 \times 3.14 \times 0.89} = 1.20 \times 10^{-3} \text{ watt/cm}^{\circ}C$$

This compares favorably with the values reported in our previous studies.[19]

2.7.2 Line Heat Source

The calculations for the line heat source apparatus are identical to those given above. A description of the curve matching method has been given in our previous work.[19]

3.0 REFERENCES

1. Black, I. A., Fowle, A. A., Glaser, P. E., "Single-Plate Apparatus for Tests of Low-Temperature Thermal Conductivity", X International Congress of Refrigeration, Paris, 1959.

2. Everest, A., Glaser, P. E., and Wechsler, A. E., "Thermal Conductivity of Non-Metallic Materials", Summary Report, Prepared by Arthur D. Little, Inc., for NASA Marshall Space Flight Center under Contract NAS8-1567, April 1963.

3. Wechsler, A. E., Glaser, P. E., and Allen, R. V., "Thermal Conductivity of Non-Metallic Materials", Summary Report, Prepared by Arthur D. Little, Inc., for NASA Marshall Space Flight Center under Contract NAS8-1567, April 1963.

4. Black, I. A. and Glaser, P. E., "The Performance of a Double-Guarded Cold Plate Thermal Conductivity Apparatus", Advances in Cryogenic Engineering, 9, Plenum Press, 1964.

5. "Liquid Propellant Losses During Space Flight", Final Report, NASA Contract No. NASw-615.

6. Schleiermacher, A. L. E. F., "Uber die Waermeleitung der Gase", Annalen der Physik U. Chemie, 34, p. 623 (1888).

7. Stalhane, B. and Pyk, S., "New Method for Determining the Coefficients of Thermal Conductivity", Teknisk Tidskift, 61, p. 389 (1931).

8. Van der Held, E. M. F. and Van Drunen, F. G., "A Method of Measuring the Thermal Conductivity of Liquids", Physica, 15, p. 865 (1949).

9. Glaser, P. E. and Kayan, C. F., "Variation of Thermal Conductivity with Temperature of Insulating Powders", Refrigerating Engineering, 64, p. 31 (1956).

10. Arthur D. Little, Inc., "Studies of the Characteristics of Probable Lunar Surface Materials", Final Report, Prepared for Air Force Cambridge Research Laboratories under Contract AF 19(628)-421.

11. Hooper, F. C. and Lepper, F. R., "Transient Heat Flow Apparatus for the Determination of Thermal Conductivities", ASHVE Trans., 56, p. 309 (1950).

12. de Vries, D. A., "A Non-Stationary Method for Determining Thermal Conductivity of Soil in Situ", Soil Science, 73, p. 83 (1952).

13. Mason, V. V. and Kurtz, M., "Rapid Measurement of Thermal Resistivity of Soil", Part 3, A.I.E.E. Trans., Power Apparatus and Systems, No. 1, p. 570-577 (1952).

14. Hooper, F. C. and Chang, S. C., "Development of the Thermal Conductivity Probe", ASHVE Trans., 59, p. 463 (1953).

15. D'Eustachio, D. and Schreiner, R. E., "A Study of a Transient Method for Measuring Thermal Conductivity", ASHVE Trans., 58, p. 331 (1952).

16. Lentz, C. P., "A Transient Heat Flow Method of Determining Thermal Conductivity: Application to Insulating Materials", Can. J. Technology, 30, p. 153 (1952).

17. Woodside, W. and Messmer, J. H., "Thermal Conductivity of Porous Media: I. Unconsolidated Sands, II. Consolidated Rocks", J. Appl. Phys., 32, p. 1688 (1961).

18. Von Herzen, R. and Maxwell, A. E., "The Measurement of Thermal Conductivity of Deep Sea Sediments by a Needle Probe Method", J. Geophys. Research, 64, p. 1557 (1959).

19. Wechsler, A. E. and Glaser, P. E., "Thermal Conductivity of Non-Metallic Materials", Summary Report, Prepared by Arthur D. Little, Inc., for George C. Marshall Space Flight Center under Contract NAS8-1567, 27 April 1964.

20. Wechsler, A. E. and Glaser, P. E., "Research Directed toward the Study of Heat Transfer in Complex Rock Systems", AFCRL 64-738, Prepared by Arthur D. Little, Inc., under Contract AF 19(628)-3279, Sept. 1964.

21. Carslaw, H. S. and Jaeger, J. C., "Conduction of Heat in Solids", Oxford Univ. Press, London (1959).

22. Blackwell, J. H., "A Transient-Flow Method for Determination of Thermal Constants of Insulating Materials in Bulk", J. Appl. Phys., 25, p. 137 (1954).

23. Blackwell, J. H., "The Axial Flow Error in the Thermal Conductivity Probe", Can. J. of Physics, 34, p. 412 (1956).

24. Wechsler, A. E. and Kritz, M., Unpublished work conducted by Arthur D. Little, Inc., for the U. S. Army Cold Regions Research and Engineering Laboratory under Contract DA 27-021-AMC-25(x), 1964.

4.0 ACKNOWLEDGMENTS

The guidance and cooperation of Mr. C. D. Cochran and Mr. J. Fountain of the Research Projects Laboratory throughout this program materially added to its successful completion.

The authors also gratefully acknowledge the assistance of Dr. P. E. Glaser and Messrs. F. E. Tilton, R. Carroll, and E. J. Boudreau during the design, construction, and testing of the apparatus. The thorough and careful technique of Mr. R. Carroll greatly facilitated the testing and installation of the apparatus.